Sampling and Analysis Plan/Quality Assurance Project Plan
2014 Residential Soil Sampling

Upper Animas Mining District
Silverton, San Juan County, Colorado

Prepared For:
United States Environmental Protection Agency, Region 8
Ecosystem Protection and Remediation – Program Support
1595 Wynkoop St.
Denver, Colorado 80202

Prepared By:
United States Environmental Protection Agency, Region 8
Environmental Services Assistance Team (ESAT)
TechLaw, Inc.
16194 W. 45th Drive
Golden, Colorado 80403

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Approval Sheet

ESAT Team Manager: _____

Signature/Date
Mark McDaniel
Region 8 Environmental Services
Assistance Team Manager

ESAT Representative: _____

Signature/Date
Steve Auer
Region 8 Environmental Services
Assistance Team Representative

EPA Remedial Project Manager: _____

Signature/Date
Paula Schmittdiel
Region 8 Remedial Project Manager

EPA Representative: _____

Signature/Date
Dan Wall
Region 8 Task Order Project
Officer/Quality Assurance Approving
Official

EPA Toxicologist _____

Signature/Date

Susan Griffin
Region 8 Toxicologist

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List of Abbreviations and Acronyms

ARSG Animas River Stakeholder Group
BLM Bureau of Land Management
CA Corrective Action
CDPHE Colorado Department of Public Health and Environment
CFR Code of Federal Register
CLP Contract Laboratory Program

cm Centimeter
COPC Contaminant of Potential Concern
DQA Data Quality Assessment
DQO Data Quality Objectives
DRMS Division of Reclamation, Mining and Safety
DS Decision Statement
EDD Electronic Data Deliverable
ESAT Environmental Services Assistance Team
EPA United States Environmental Protection Agency
gpm Gallon per minute
GPS Global Positioning System
HRS Hazard Ranking System
LCS Laboratory Control Spikes
LCSD Laboratory Control Spike Duplicates
LIMS Laboratory Information Management System
mL Milliliter
MS/MSD Matrix Spike/Matrix Spike Duplicate
NPL National Priorities List
OSHA Occupational Safety and Health Administration
PE Performance Evaluation
PSQ Principal Study Question
QA/QC Quality Assurance/Quality Control
QAO Quality Assurance Officer
QAPP Quality Assurance Project Plan
QMP Quality Management Plan
RPD Relative Percent Difference
RPM Remedial Project Manager
SAM Site Assessment Manager
SAP Sampling Analysis Plan
SAR Sampling Activities Report
SGC Sunnyside Gold Corporation
SOP Standard Operating Procedures
Sq. ft. Square feet
TAL Target Analyte List
TOPO Task Order Project Officer
USFS United States Forest Service
USFWS United States Fish and Wildlife Service
USGS United States Geological Survey
XRF X-Ray Fluorescence

A.3 Distribution List

The following is a distribution list of personnel that will receive a copy of the Sampling and Analysis Plan (SAP)/Quality Assurance Project Plan (QAPP) for the residential and municipal soil sampling event scheduled in 2014 that will be conducted in the Town of Silverton, Colorado. Agency and contractor affiliations are also listed for each individual.

Paula Schmittdiel EPA
Victor Ketellapper EPA
Dan Wall EPA
Nicole Plescia EPA
Susan Griffin EPA
Sabrina Forrest EPA
Steven Auer ESAT Field Task Lead
Mark McDaniel ESAT Manager
Doug Jamison CDPHE
Mayor & City Council City of Silverton
Board of Commissioners San Juan County

A.4 Project/Task Organization

The following is a list of involved personnel, respective agency, contract affiliation, and general responsibilities.

Managers:

Paula Schmittdiel EPA Remedial Project Manager
Victor Ketellapper EPA Site Assessment Team Leader
Dan Wall EPA Task Order Project Officer/
QA Approving Official
Susan Griffin EPA EPA Toxicologist
Mark McDaniel ESAT ESAT Team Manager

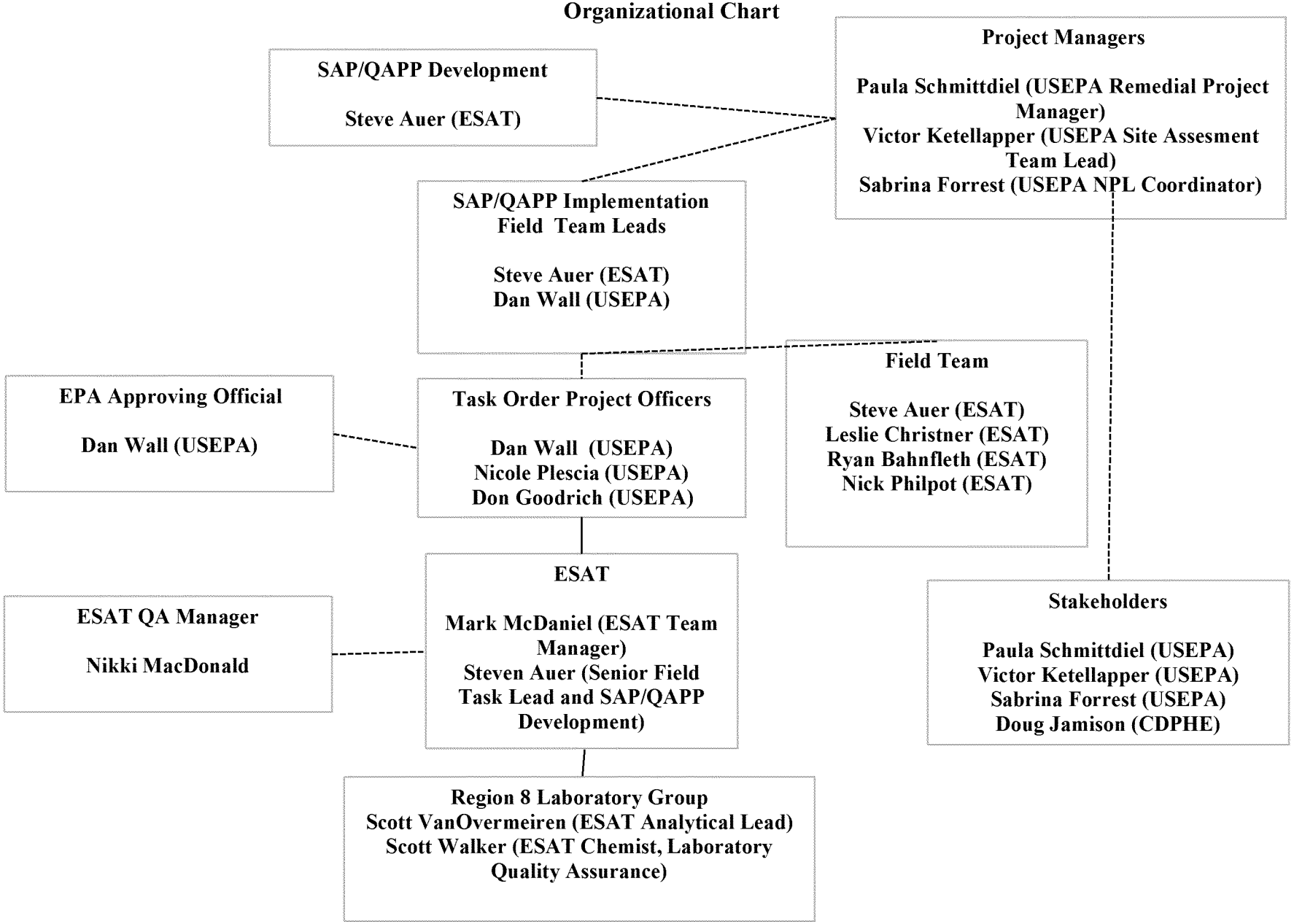
Field Team:

Steve Auer ESAT Field Task Lead
Ryan Bahnfleth ESAT Field Support
Nick Philpot ESAT Field Support
Leslie Christner ESAT Field Support

Laboratory Group:

Scott VanOvermeiren ESAT Inorganic Task Lead
Scott Walker ESAT Analytical Support

Organizational Chart



A.5 Problem Definition

Silverton is a former silver mining camp located in San Juan County, in the southwest region of Colorado. Historical mining, milling and smelting activities may have resulted in soil contamination from metals such as arsenic, cadmium, lead, zinc, barium, and mercury. The United States Environmental Protection Agency (EPA) site assessment program needs additional data to determine if historical mining, smelting, milling or concentrating operations in Silverton and the surrounding area have resulted in metals contaminated residential soils that may pose a threat to human health or the environment. Specifically, the goals of these investigations are to:

- Collect samples of fine-grained (<2mm) slag material, other sources, and soils located onsite to determine if they are a source of contamination;
- Collect surface and subsurface soil samples from residential yards, municipal grounds, school yards, and playgrounds in the neighborhoods surrounding the former Silverton smelters sites to determine metals concentrations and to identify areas of observed contamination associated with the site;
- Collect background soil samples for the purpose of comparing metals concentrations in background samples with residential soil samples and applicable health-based benchmarks for evaluation using the Hazard Ranking System (HRS);
- Support the remedial and removal programs in the assessment of unacceptable risks to residential receptors via the soil exposure pathway.

A.5.1 Introduction

The Environmental Services Assistance Team (ESAT) in conjunction with EPA will be collecting soil samples throughout the Town of Silverton, Colorado in order to characterize sources and residential and municipal soils that may have been impacted by historical mining operations in the area. Sampling is expected to occur in two stages during the 2015 sampling season and will occur at residences where access is granted, as well as municipal areas including (but not limited to) municipal grounds, school yards, and playgrounds. Overall it is anticipated that 50-75 residential yards and ten parks, schools or other public areas will be sampled as a part of this investigation. Five to six individual source or slag samples will be also be collected to characterize source areas. Approximately three background soil samples will be collected to establish background conditions in the area. Finally, a total of ten surface soil samples and one to two source or slag samples will be collected and analyzed to establish in-vitro bioaccessibility.

This SAP has been prepared in accordance with the EPA *Guidance on Systematic Planning Using the Data Quality Objectives Process (EPA QA/G-4)*, *Requirements for Quality Assurance Project Plans (QA/R-5)*, *Superfund Lead-Contaminated Residential*

Sites Handbook for the delineation of sampling zones, sampling number and depth (http://www.epa.gov/superfund/lead/products/handbook.pdf), and the *Guidance for Quality Assurance Project Plans (EPA QA/G-5)*, (EPA 2006; EPA 2001; EPA 2003; EPA 2002). This SAP is designed to guide field work that will include the collection of soil samples as well as associated field Quality Assurance/Quality Control (QA/QC) samples.

Field activities will include performing in-situ X-Ray Flourescence (XRF) spectrophotometric analysis on soil samples. Twenty percent (20%) of the in-situ XRF field samples will then be properly prepped and resampled with XRF before and then sent to either the Region 8 ESAT lab or CLP lab for collaborative testing procedures. Laboratory analyses of soil samples will include total recoverable Target Analyte List (TAL) metals. Prior to conducting XRF analysis on all soil samples, preliminary analysis of ten soil samples will be conducted by both XRF and in the laboratory to verify Contaminants of Potential Concern (COPCs) and to determine if inter-analyte interference is going to be a problem at the site which might preclude the use of XRF on a larger scale. All samples will be analyzed either directly through the ESAT Region 8 Laboratory or indirectly (i.e., submitted through a CLP laboratory) or analyzed in the field by XRF.

Sampling procedures will adhere strictly to those outlined in the ESAT Region 8 Standard Operating Procedures (SOPs) and the *Superfund Lead-Contaminated Residential Site Handboo*k (EPA 2003). Deviations from the SAP will be documented in the site-dedicated field notebook and reported in the Sampling Activities Report (SAR). Deviations that result in major modifications to the SAP will be noted and incorporated into all addenda to the SAP, which will be followed as applicable for subsequent sampling events. A brief addendum to the SAP will be created for each subsequent sampling event by updating relevant figures, tables, and attachments. Data obtained from these investigations will be used in accordance with the provisions outlined in the Data Quality Objectives (DQOs).

A.5.2 Background

The history of mining production activities in and around Silverton, Colorado and the San Juan County spans more than 120 years and began in the early 1870s when gold and silver was discovered in Silverton and the Animas Mining district and brought an influx of miners. The discovery of silver in the base-metal ores was the major factor in establishing Silverton as a permanent settlement. Between 1870 and 1890, the richer ore deposits were discovered and mined to the extent possible. Not until 1890 was any serious attempt made to mine and concentrate the larger low-grade ore bodies in the area.

By 1900, there were twelve concentration mills in the valley sending products to the Kendrick and Gelder Smelter near the mouth of Cement Creek. Mining and milling operations slowed down circa 1905, and mines were consolidated into fewer and larger operations with facilities for milling large volumes of ore. The Rose Walsh smelter operated in Silverton and was located at the "western" edge of Silverton. This smelter was operational for four years and was eventually shut down in 1897. After 1907, mining and milling continued throughout the basin whenever prices were relatively favorable.

Silverton is located on the Animas River at the confluence with Cement Creek and is a former silver mining camp settled in 1874, a center for historical mining activities in the surrounding area. The town was the central location and railroad terminus for the milling and shipping of mine ores. The town declined in the 1920s and today has less than 500 residents. By the 1970's only one year-round productive mine (Sunnyside Mine) remained in the county. This mine ceased production in 1991, and has since undergone extensive reclamation efforts. The Gold King Mine's permit with the Division of Reclamation, Mining and Safety (DRMS) is currently inactive; however, landowners hope to rehabilitate the mine. In 2003, the location of the former Rose-Walsh Smelter was selected for development under a Brownfield Assessment.

A Targeted Brownfields Assessment was conducted by EPA in 2005 which found levels of lead and arsenic up to 34,000 ppm. A Voluntary Cleanup of that site was performed by the State in preparation for the development of affordable housing in Silverton.

Today, a number of abandoned mines and structures from the late 1800s through the 1900s exist in and around Silverton, such as the North Star (Sultan) Mill structure located along the western edge of Silverton, along Mineral Creek. Indeed, historically, there were over 30 mines, mine tailings, mills, and smelters within a two mile radius of Silverton (see Table A.5-1).

EPA along with ESAT, United States Fish and Wildlife Service (USFWS), and United States Geological Service (USGS) performed three sampling events from 2012 to 2013 to add to previously collected data in order to further understand the nature and extent of contamination to the surface water due to historical mining activities. Over the three events, surface water, sediment, macroinvertebrates, stream flows and real-time water quality parameters were collected. However, to date no assessment has been performed to characterize metals concentrations in soils within the Town of Silverton, specifically in residential yards, municipal grounds, school yards, playgrounds or other public areas. This effort will help determine if metals in soils are present at concentrations above

background and health-based benchmarks and if they are associated with historical mining operations.

A.6 Project/Task Description

Sampling will be conducted in 2015 to characterize metals levels in residential soils as well as in soils associated with public areas (such as municipal grounds, school yards and playgrounds) in the Town of Silverton. Data generated from the sampling events will be used in accordance with the established DQOs (Section A.7.2) and will be evaluated using the HRS. The following data will be collected during the events:

- Surface soil samples for in-situ XRF analysis
- Surface soil samples for laboratory of total recoverable metals
- Subsurface soil samples for in-situ XRF analysis from depths of 0-6", 6-12", and 12-18"
- Subsurface soil samples for laboratory analysis of total recoverable metals from depths of 0-6", 6-12", and 12-18"
- Slag or source soil samples for in-situ XRF analysis
- Slag or source soil samples for laboratory analysis of total recoverable metals
- Discrete soil samples (not tailings or rock waste) from locations expected to be biased high and located within the property boundaries of a residence, school, or park within 200 feet of a building or active school playground
- Background soil samples for total recoverable metals will be sent for laboratory analysis
- Surface soil samples sieved to 250 micron particulate size for evaluation of in-vitro bioaccessibility of lead and arsenic
- Slag or source samples for evaluation of in-vitro bioaccessibility of lead and arsenic

A.7 Quality Objectives and Criteria

This section discusses the DQO process and how it was applied to this study. Specific areas addressed include: the planning team and stakeholders, DQOs, and parameter metrics such as precision, accuracy, representativeness, completeness, comparability and sensitivity. Data collected during these sampling events will be intended to achieve the following:

- Collect surface and subsurface soil samples from residential yards, municipal grounds, school yards, and playgrounds in the neighborhoods surrounding the former Silverton smelters sites to determine metals concentrations and to identify areas of observed contamination associated with the site. Collect background soil samples for the purpose of comparing metals concentrations in background samples with residential soil samples and applicable health-based benchmarks for evaluation using the HRS;

- Support the remedial and removal programs in the assessment of unacceptable risks to residential receptors via soil exposure pathways.

A.7.1 Planning Team and Stakeholders

The following sections list the members of the DQO planning team, primary decision makers, and parties who may be impacted by the results of this study or who may use the data generated as a result of the DQO process.

A.7.1.1 DQO Planning Team

The following table includes the DQO planning team members, respective organizations, and affiliation with that organization.

Table A.7-1 DQO Planning Team

Name	Organization	Area of Technical Expertise
Paula Schmittdiel	EPA Region 8	Remedial Project Manager
Dan Wall	EPA Region 8	Task Order Project Officer
Victor Ketellapper	EPA Region 8	Site Assessment Team Leader
Susan Griffin	EPA Region 8	Toxicologist
Sabrina Forest	EPA Region 8	NPL Coordinator
Steve Auer	ESAT	Biologist

A.7.1.2 Decision-Making Authority

The decision maker has the ultimate authority for making final decisions based on the recommendations of the DQO team. The decision makers for this event are Victor Ketellapper, EPA Site Assessment Manager (SAM) and Paula Schmittdiel, the EPA Region 8 Remedial Project Manager (RPM) for this site.

A.7.1.3 Stakeholders

Stakeholders are parties who may be affected by the results of the study or persons who may later use the data resulting from the DQO process. Table A.7-2 lists the impacted organizations and stakeholders, and the individuals that are representing those organizations.

Table A.7-2 Stakeholders

Organization	Represented By
EPA Region 8	Paula Schmittdiel
EPA Region 8	Victor Ketellapper
EPA Region 8	Sabrina Forrest
Colorado Dpt. of Public Health & Environment	Doug Jamison

A.7.2 Data Quality Objectives

The DQO process specifies project decisions, the data quality required to support those decisions, specific data types needed, data collection requirements, and analytical techniques necessary to generate the specified data quality. The process also ensures that the resources required to generate the data are justified. The DQO process consists of the following seven steps:

1. State the problem,
2. Identify the goal of the study,
3. Identify the information inputs,
4. Define the boundaries of the study,
5. Develop the analytic approach,
6. Specify performance or acceptance criteria, and
7. Develop the plan for obtaining data.

During the first six steps of the process, the planning team develops decision performance criteria that will be used to develop the data collection design. The final step of the process involves developing the data collection design based on the DQOs. A brief discussion of these steps and their application to this project are provided in the following sections.

A.7.2.1 Step 1: State the Problem

The Town of Silverton, Colorado is a former silver mining camp in which milling and smelting activities in the past may have resulted in soil contamination within town limits. The first recorded mill in the Silverton camp was the Little Giant, established in 1873. The first smelters followed the following year. During smelter operations, heavy metal contaminants were likely exhausted through the smoke stacks and dispersed through the air pathway to surficial soils. Inhalation of windblown soils historically contaminated with lead and arsenic may still pose a risk to human health. Fine-grained source and soil particulates contaminated with heavy metals may pose an ongoing risk to human health via inhalation. Evaluation of the air pathway is being deferred until after the collection and evaluation of residential soils. If the results of analysis of residential soils indicate unacceptable concentrations of heavy metals, the air pathway may be investigated. Contamination from inorganic metals such as lead, zinc, arsenic, cadmium, barium, and mercury are typical with the types of mining, milling, and smelting activities historically conducted in the Silverton area. The current impact of these activities on residential and public area soils is not well understood; however, smelting and mining activities have been associated with aerial deposition via smoke stacks and the transport of slag materials within residential areas. Direct exposure with contaminated soil and potential inhalation of fine-grained windblown contaminant particles may pose an ongoing risk to the local population. Additional data are needed to determine if the residential as well as

public area soils (such as municipal grounds, school yards and playgrounds) have been impacted by historical mining activities and associated source areas. Additionally, data are needed to compare to applicable health-based benchmarks, background levels, and to support the remedial and removal programs in the assessment of unacceptable risks to residential receptors. A site conceptual exposure model is provided in Figure A.7-1 and shows an evaluation of contaminant sources, release mechanisms, exposure media, exposure routes, and potential receptors.

A.7.2.2 Step 2: Identify the Goals of the Study

The purpose of this step is to define the principle study questions (PSQs) that this study will attempt to resolve. The PSQs will help determine appropriate data inputs and potential alternative actions. Principle study questions can be used to develop decision statements (DSs) when the potential alternative actions have been determined to resolve the problem. In situations where the outcomes may not lead to specific decisions or the information may be used to gain a greater understanding of existing data, estimation statements are more appropriate. Estimation statements are more applicable to the nature of the PSQs being investigated in these sampling efforts.

The PSQs are as follows:

PSQ1 – What are the levels of metals concentrations in surface and subsurface soils in residential yards, municipal grounds, school yards and playgrounds?

PSQ2 – What are the levels of metals concentrations in mining related source or slag samples?

PSQ3 – What are the levels of metals in background soil samples?

PSQ4 – Do metals concentrations in surface and subsurface soil samples exceed applicable health-based benchmarks?

PSQ5 – Do metals concentrations in surface and subsurface soil pose an unacceptable risk to human receptors?

Estimation Statements

PSQ1 – What are the levels of metals concentrations in surface and subsurface soils in residential yards, municipal grounds, school yards and playgrounds?

Analytical results of surface and subsurface soil samples (both laboratory and in-situ) will be used to determine the level of metals concentrations in residential yards, municipal grounds, school yards, and playgrounds.

PSQ2 – What are the levels of metals concentrations in mining related source or slag samples?

Analytical results of surface and subsurface soil samples (both laboratory and in-situ) will be used to determine the level of metals concentration in areas identified as associated with historical mining activities, such as mining source areas, tailings, and slag areas.

PSQ3 – What are the levels of metals in background soil samples?

Analytical results from background will be used to determine the level of metal concentrations in the ambient background environment. These results will be compared to source and residential sampling results to determine if source and residential sampling results are above the ambient background environment.

PSQ4 – Do metals concentrations in surface and subsurface soil samples exceed applicable health-based benchmarks?

One aspect of this SAP is to determine if there is contamination present in residential soils at levels above background, if these concentrations exceed health-based benchmarks or regulatory standards, and whether they meet observed release criteria. The SAP will also determine the exact locations of these contaminants in relation to residences, the number of residences occupied and the frequency of occupation, and determine the same for schools, municipal grounds, and playgrounds in the study area.

PSQ5 – Do metals concentrations in surface and subsurface soil pose an unacceptable risk to human receptors?

Results of metals concentrations from analysis of surface and subsurface soil samples will be evaluated using HRS criteria and may be used conjunction with bioaccessibility results to determine if there may be unacceptable risks to human receptors via the given soil exposure pathway.

A.7.2.3 Step 3: Identify Information Inputs

The purpose of this step is to identify the data required to answer the PSQs listed in section A.7.2.2. The primary information and decision inputs will be data generated from field instruments and laboratory analyses.

Field parameters and non-sampling objectives include:

- Collecting XRF analytical data
- Photographing sample locations and other notable observations in a field narrative logbook
- Collecting geospatial data for sample locations
- Collecting historical information about the smelters, the number and height of any smoke stacks, and other possible mechanisms of contaminant transport

Analytical laboratory parameters for soil samples will include:

- Total recoverable metals in surface and subsurface soil

Further laboratory parameters for ten soil samples will include:

- Analysis for in-vitro bioaccessibility in surface soil samples and source or slag samples

The following factors will be evaluated in the overall decision-making process:

- Use of analytical results (in-situ or laboratory) to determine if residential yards and public areas have been impacted by source areas
- Use of analytical results (in-situ or laboratory) by the site assessment program
- Incorporation of analytical results (in-situ or laboratory) into an analytical results report which evaluates sources, the soil exposure pathway, or other appropriate pathways of the HRS.

A.7.2.4 Step 4: Define the Boundaries to the Study

The objective of this step is to define the spatial and temporal components of the study area. The scale of the decision making for the DS is defined by combining the population of interest with the spatial and temporal boundaries of the site. Practical constraints that could interfere with sampling are also identified. Implementing this step helps ensure that the data are representative of the population.

A.7.2.4.1 Spatial Boundaries

The study area boundary comprises residential properties and public areas within the town of Silverton potentially impacted by contaminant source areas associated historical mining, milling, and smelting activities.

A.7.2.4.2 Temporal Boundaries

The preliminary sampling event will take place in the Spring of 2015 at the earliest; the larger sampling event will take place in Summer and Fall 2015 depending on the availability of access. During the preliminary sampling event approximately ten samples will be collected from residential and municipal areas to prove out the sampling process and evaluate XRF performance in the field. Additionally, three to five samples will be collected from source areas—areas identified as associated with historical activities or slag areas. The remainder of the samples will be collected during the second phase of planned activities.

A.7.2.5 Step 5 : Develop the Analytic Approach

Results from in-situ or laboratory metals analysis of surface and subsurface soil samples will be used to determine if residential yards, municipal grounds, school yards or playgrounds have been contaminated by metals from source areas related to historical mining, milling, and smelting activities in and around Silverton, Colorado. As indicated above, sampling will be conducted in two stages. The preliminary stage is intended to implement and prove out the sampling approach and evaluate XRF performance in the field (as discussed below). Based on the results of the preliminary stage, the remainder of the samples will be collected in the second phase.

A.7.2.6 Step 6 : Specify Performance or Acceptance Criteria

The purpose of this step is to specify the tolerable limits on decision errors, which are used to establish performance goals for the data collection design. For this project, the number of samples is based on the number of residential properties and applicable public areas (such as municipal grounds, school yards, the predominantly unpaved streets, and playgrounds) located in the study area encountered laterally from suspected source areas, as indicated in the *Superfund Lead-Contaminated Residential Site Handbook* (EPA 2003).

In order to mitigate the potential for false positive or false negative errors associated with field sampling, sample collection processes will be consistent with established and relevant SOPs. This includes collection of duplicate samples (and subsequent analysis using relative percent difference (RPD) statistics) and implementing a decontamination procedure (which may include the use of disposable sampling equipment). During the preliminary sampling event, approximately ten soil samples (including source samples) will be collected and subsequently analyzed both by XRF and in the laboratory to verify

the COPCs and to determine if interference, such as an inability to reliably use the XRF at the site or because time constraints limit the ability to achieve temporal goals. Based on the results of the preliminary sampling event, the XRF may be used as the primary analytical tool to establish metals concentrations in samples collected during the second sampling event. To confirm the XRF data, twenty percent of all XRF-analyzed samples will be sent to the ESAT Region 8 or CLP laboratory for total recoverable metals analysis. For laboratory analysis of samples, QA/QC steps (such as the use of laboratory controls, MS/MSDs, blanks, etc.) will be consistent with ESAT Region 8 or CLP reporting requirements.

A.7.2.7 Step 7 Develop Plan for Collecting Data

The sampling design employed for these investigations is based on guidance provided in the *Superfund Lead-Contaminated Residential Site Handbook* (EPA 2003). Per that guidance, sample locations were chosen based on lateral proximity of residential and municipal properties from suspected source areas. Note that residential and municipal soils may present a direct exposure pathway to persons working, playing, or conducting other recreational activities on a property. Surface and subsurface locations will be collected, and an evaluation of in-vitro bioaccessibility of metals in surface soils and slag or source material will also be conducted. As indicated above, sampling will be conducted in two stages. The preliminary stage is intended to prove out the overall sampling approach and evaluate XRF performance in the field (approximately ten residential/municipal soil samples and three to five source/slag samples), with all remaining samples collected and analyzed as a part of the second stage. This approached is based on the assumption of uniform access to residential properties. Without uniform access to residential properties the first phase of sampling activities may be limited publicly accessible properties.

A.7.2.8 Sampling Locations

For the proposed investigations, the number of samples is based on the number of residences and municipal properties (such as municipal grounds, school yards and playgrounds) located in the study area encountered laterally from suspected source areas, as suggested in the *Superfund Lead-Contaminated Residential Site Handbook* (EPA 2003). A total of 50-75 homes and approximately ten parks, schools or other public areas are anticipated to be sampled. Additionally three background soil samples and five to six source or slag samples will also be collected. Finally, ten surface soil and one to two source or slag samples will be collected and evaluated for in-vitro bioaccessiblity. Sampling locations, descriptions, and activities that will take place are listed in Table A.8-4. Note that these locations could change based on encountered access issues or site conditions identified while in the field (e.g., access to private properties or presence of slag-impacted areas on residential properties) as well as any opportunistic samples that may be encountered.

A brief description of the sampling locations will be recorded in the site-dedicated field notebook for each residence and municipal property sampled. Information will consist of a sampling location identification number, address, property owner and any renter, date, time, access information, geographical observations, and any other pertinent information that will be useful in identifying the sampling location in the future. A photolog will also be kept to record sampling locations. Photo identifications as assigned automatically by the camera software will be logged in the site-dedicated field notebook. If there are deviations from this SAP/QAPP or applicable SOPs, including the decision to not sample a location because conditions are either unsafe or inaccessible, these changes will be recorded in the site-dedicated notebook, as will the addition of any opportunity samples.

A.7.3 Precision, Accuracy, Representativeness, Completeness, Comparability, and Sensitivity

The documentation of the data evaluation effort will be in the form of the worksheets prepared during validation. These worksheets will be an appendix to the SAR. The SAR will be prepared to identify problems that may affect data usability or require that the data be qualified. The SAR report will discuss all precision, accuracy, representativeness, completeness, comparability, and sensitivity parameter results from the data validation and overall usability of the data for project objectives, including the following:

Precision:
- Field duplicates: RSD criteria met?
- Laboratory duplicates: RSD criteria met?
- Method of standard dilution performed and criteria met?
- MSDs: RPD criteria met? (If applicable)

Accuracy:
- MS/ MSDs: percent recovery (%R) criteria met?
- Laboratory control sample (LCS)/laboratory control sample duplicates (LCSDs): %R criteria met?
- Initial and continuing calibration recoveries met?
- Interference check sample recoveries met?
- Inductively coupled plasma serial dilution recoveries met?

Representativeness:
- Sampling procedures and design: criteria met?
- Holding times and preservation: criteria met?
- Custody: all chain-of-custody forms complete and provided in data package?
- Blanks: contaminants present?

Completeness

- The number of valid analytical results is comparable (90%) with the number determined necessary during establishment of DQOs.

Comparability:

- Data compares with similar analysis and data sets?
- Sample collection methods comparable to similar data sets?
- Laboratory analytical methods comparable to similar data sets?

Sensitivity:
Method reporting limits met project objectives?

The data will be assessed for the following criteria:

- *Bias* – a systematic or persistent distortion of a measurement process that causes errors in one direction. The extent of bias will be determined by evaluating the laboratory initial calibration and continuing calibration verification, LCS/LCSD, blank spikes, MS/MSD, and method blanks.
- *Sensitivity* – the ability to discriminate between small differences in analyte concentration is related to the rate of change in response when there is a small change in stimulus and is reflected in the calibration curve. The detection limits of the field and laboratory methods are within the range of previous detections found at the site.
- *Precision* – the measure of agreement among repeated measurements of the same property under identical, or substantially similar, conditions and which is expressed as the RPD between the sample pairs. An acceptable RPD for soil is 35% (EPA, 2010).
- *Representativeness* – the measure of the degree to which data accurately and precisely represent a characteristic of a population parameter, variations at a sampling point, a process condition, or an environmental condition.
- *Completeness* – a measure of the amount of valid data obtained from a measurement system. The actual percentage of completeness is less important than the effect of completeness on the data set. Completeness will be assessed by the total number of samples collected versus the amount of samples planned.
- *Comparability* – the qualitative term that expresses the confidence that two data sets can allow for common interpretation and analysis; comparability is used to describe how well samples within a data set, as well as two independent data sets, are interchangeable.

Uncertainty of validated data will be evaluated by the RPM and the EPA SAM or their designees to determine if the DQOs were met. In the event that the DQOs were not met, they will be reviewed to determine if they are achievable and may be revised if necessary, and the data may be further evaluated to determine the impact to the project. Data usability and limitations will be evaluated by the RPM and EPA SAM.

As discussed above, all samples will be analyzed either at the ESAT Region 8 or CLP laboratory, or in-situ via XRF. If samples are analyzed using XRF, 20% will be sent for laboratory analysis of metals for collaborative purposes. Prior to conducting XRF analyses of all soil samples, a preliminary sampling and analysis of approximately ten soil samples will be conducted by both XRF and laboratory to verify COPCs and determine whether interference will be a problem at the site.

A.8 Special Training/Certifications

All field staff have completed the Occupational Safety and Health Administration (OSHA) 40-hour Health and Safety Course for Hazardous Waste Site Worker Training in accordance with Sections e and p of OSHA 29 Code of Federal Regulation (CFR) 1910.120 and maintain this certification with annual eight-hour Hazardous Waste Site Operations Refresher Training as required by Sections e and q of OSHA 29 CFR 1910.120. Field staff have completed American Red Cross Standard First Aid and Adult CPR Training and maintain this certification annually for Adult CPR and every two years for Standard First Aid. The ESAT and EPA Health and Safety Managers are responsible for ensuring that all field staff complete the training requirements as required by OSHA.

A.9 Documentation and Records

Field measurements will be recorded in a site-dedicated field notebook at the time of data collection. The data sheets from the data download will be printed, and scanned copies will be included in the SAR. Field notebooks, chain-of-custody forms and other forms used for the site investigation will be stored at the ESAT Region 8 EPA Laboratory Suite A127.

The documentation of the data evaluation efforts will be in the form of the worksheets prepared during validation. These worksheets will be an appendix to this SAP and will be provided in a subsequent SAR. The SAR will be prepared to identify problems that may affect data usability or require that the data be qualified. The SAR will discuss all precision, accuracy, representativeness, completeness, comparability, and sensitivity parameter results from the data validation and overall usability of the data for project objectives.

The laboratory will submit to EPA a data report containing all the analytical results for this sampling effort. The report will contain a case narrative that briefly describes the

number of samples, analyses, and any analytical difficulties or QA/QC issues associated with the samples. The data report will also include signed chain-of-custody forms, analytical data, a QA/QC package, and raw data. Additional reporting requirements are outlined in the ESAT laboratory contract and quality management plan (QMP).

Peer review of the data package, at a 100% frequency of reported versus raw data, will be performed by the analytical laboratory. The final report of the abbreviated data validation will be in a standard Stage 4 Contract Laboratory Program (CLP) format, including all laboratory and instrument QC results.

B. DATA GENERATION AND ACQUISITION

This section describes data generation and acquisition activities associated with these events, including process design, sampling and analytical methods, sample handling and custody, QC, equipment, and data use and management.

B.1 Sampling Process Design

The following sections describe the sampling methods to collect soil samples for inorganics analysis and in-vitro bioaccessibility. Appendix A provides copies of the applicable SOPs, outlining how field activities will be performed (including documentation protocols). Tables B.1-1 and B.1-2 provide the sampling checklist and field equipment checklist, respectively. Section A.7 provides the rationale for the sampling process outlined in this section.

The EPA RPM, or the EPA SAM or the SAM designee will be responsible for directing corrective actions if problems are encountered in the field which would impact the way this SAP/QAPP is implemented, or if sampling locations are inaccessible. Dan Wall is the EPA designee appointed for the studies. Any problems encountered and actions taken or deviations from this SAP/QAPP will be documented in the field notebook.

B.1.1 Residential and Municipal Surface and Subsurface Soil Sampling

As per the *Superfund Lead-Contaminated Residential Site Handbook* (EPA 2003), since the source areas of contamination include former mines, mills, and smelters, variability in metals concentrations is expected. Since this variability is expected, the EPA Guidance suggests that every home in the potentially affected area be sampled, moving laterally away from the source until clean areas of the community have been identified. This general approach will be followed for residential and municipal properties alike, given uniform site access. Uniform site access is assumed in the sampling approach presented. Without uniform site access, this SAP may have to be altered in order to account for limited access. This issue will be addressed by regulators and stakeholders prior to site activities.

B.1.2 Nature of Data Collected

As indicated in Section A.7, a variety of data will be collected during these events, some of which are critical to achieve the established DQOs and project objectives, and some of which are primarily for informational purposes or which will be used to supplement critical data. The following chart specifies each type:

Data Type	Purpose
Residential/municipal surface soil samples	Critical
Residential/municipal subsurface soil samples	Critical
Source or slag discrete soil samples	Critical
Background soil samples	Critical
Geospatial data	Critical
Photolog	Informational
General field observations noted in logbook	Informational

B.1.3 Data Variability

To help assess data variability of field samples, field QC samples are collected. These field QC samples include duplicates and XRF confirmatory samples. Twenty percent of samples analyzed with an XRF device will be sent to the lab to confirm the XRF results. For every one in ten samples a field duplicate will be collected. Additionally, during the preliminary sampling event, ten soil samples will be collected and analyzed by XRF and at the laboratory to verify COPCs and determine if interference is going to be a problem that might preclude XRF use in the field.

B.2 Sampling Methods

This section describes residential soil sampling of source and background area methods that will be employed during these sampling events as well as the applicable SOPs, necessary equipment, and support facilities.

B.2.1 Equipment and Support Facilities

Specific field equipment necessary for execution of the SAP is included in Table B.1-2. During field activities, it is anticipated additional support facilities and vehicles outside of the sampling vehicle will not be needed.

B.2.2 Surface and Subsurface Soil Sampling

For residential homes less than 5,000 square feet (sq ft) in surface area, a five-point composite sample will be collected from each the front yard, back yard and side yard at zero to two centimeters (cm). Soil from each aliquot will be collected in a plastic bag, and then a portion of each aliquot will be placed in an appropriate container, such as a stainless steel bowl or a plastic bag, and thoroughly mixed. The individual aliquots will be saved and sent to the Region 8 laboratory for confirmatory analysis of the infield XRF

sampling results. If the surface area is greater than 5000 sq ft, the area will be divided into four equally-sized quadrants and a five-point composite (0-2 cm) will be collected from each quadrant. These samples should be outside the drip zone (described below) and away from any painted surfaces. In addition to the surface soil samples, subsurface samples will be collected from one location on each property at increments of zero to six inches, six to twelve inches, and twelve to eighteen inches to determine if contamination is present at the given depth.

Rooftops may collect fine-grained sediments that contain high concentrations of lead. In areas where downspouts discharge during a storm event, the fine-grained material washed from a roof may accumulate and result in a localized increase in lead concentrations in the soil. These areas are called drip zones, which are defined as six to thirty inches from the exterior walls of a building. Drip zones that are encountered will be sampled using a four-point composite sample methodology with one sample collected from each side of a residential unit, regardless of surface area.

The sampling process will consist of discrete aliquots of equal amounts of soil. In some cases, material other than grass or soil will be encountered at a sample location (e.g., wood chips and sand that are often found in recreational areas of day-care and school playgrounds). Samples of the soil below the cover material should be collected. The soil from each aliquot will be collected using a disposable plastic trowel, thoroughly mixed, and placed into one clean container such as a stainless steel bowl. Samples should be dried at a low temperature and sieved to <250 microns (#60 mesh) before being analyzed by XRF. Twenty percent of XRF-tested samples will be sent to the ESAT Region 8 or CLP laboratory in a 4 oz. glass jar. These samples will be subsampled by the field team in the same fashion that the XRF sample was prepped. The sampled aliquots will be retained and preserved for confirmatory laboratory analysis. Any remaining sample volume can then be disposed of at the general location from where it was collected. Soil mixing and sieving equipment will be decontaminated between each sample. Decontamination will include the following: initial wash with a ten percent liquinox or equivalent non-phosphate-based biodegradable detergent followed by a deionized water rinse.

An additional ten discrete soil samples (not tailings or rock waste) will be collected from locations expected to be biased high and located within the property boundaries of a residence, municipal grounds, school yard, or playground within two hundred feet of a building or active playground. Approximately five to six source or slag samples and three soil background samples will also be collected. All of the biased, source or slag, and background samples will be sent to the ESAT Region 8 or CLP laboratory for analysis. Approximately ten to twelve surface soil samples will be selected for analysis of in-vitro

bioaccessibility of lead and arsenic with potentially two of those being source or slag samples. Sampling locations will be selected by the EPA RPM and the EPA SAM, and will be based on access to the properties.

B.3 Sampling Handling and Custody

Sample designations will consist of a series of letters and numbers to indicate site and location. Table A.8-4 shows the naming convention used and site descriptions. The sampling areas are as follows:

BG - Background
SS - Source or Slag
PA - Public Area
R - Residence

All samples will be preserved and sent to the ESAT Region 8 laboratory following chain-of-custody protocols. The samples will be relinquished to the sample custodian at the laboratory, together with the completed and signed chain-of-custody forms. Attachment B provides a blank chain-of-custody form. The sample custodian will inspect the coolers upon arrival to make sure that the proper temperature was maintained, that the sample containers are intact and sealed, and that the number of samples in the coolers match the information provided in the chain-of-custody forms. All the samples will be stored in an access-controlled sample cooler at the laboratory. An analytical chemist will log the samples in the Laboratory Information Management System (LIMS) upon receipt and will enter all analytical data into the SCRIBE database for permanent storage and archiving.

All field measurements and observations will be recorded in a bound notebook or on appropriate data sheets by the field personnel at the time they are performed in accordance with the Contract Laboratory Program Guidance for Field Samplers (EPA, 2011). The personnel doing the recording will initial and date all measurements, observations, and any other notations made. Corrections will be performed by drawing a single line through the error accompanied by the date and the initials of the person performing the correction, followed by the proper entry. Chain-of-custody forms will be filled out during the time of collection and will follow the protocol provided in *Sample Custody and Labeling,* SOP# FLD-11 (ESAT, 2012).

B.3.1 Soil Sample Preservation

After soil samples have been dried at a low temperature, sieved, and sampled with the XRF soil samples selected for laboratory analysis will be sealed in pre-cleaned 125

milliliter (mL) EPA approved glass containers. Soil samples can be held for six months; no ice or preservative is needed for preservation.

B.4 Analytical Methods

Samples will be analyzed for total recoverable metals as well as in-vitro bioaccessibility. Table A.7-3 includes the laboratory analytical instrumentation and methods to be used for sample analysis. These methods will be in accordance with EPA *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, also known as SW-846, Method 7473, Revision 0, January 1998. Additionally, sample analysis will be in accordance with Method 200.7 *Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry*, Revision 4.4, May 1994, Method 200.8 *Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Mass Spectrometry*, Revision 5.4, May 1994, and Method 6200 *Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment,* Revision 0, February 2007.

Sample disposal of potentially hazardous waste will follow protocol defined in *Collection, Analysis and Disposal of ESAT Laboratory Waste* SOP LAB01.01 (ESAT, 2012).

In the event that problems are encountered in the field that may impact the implementation of this SAP/QAPP, the EPA RPM, the EPA SAM, or their designees will be responsible for directing corrective actions. The designees appointed for these events are Dan Wall and Susan Griffin. Any problems encountered and actions taken or deviations from this SAP will be documented in the field notebook and stakeholders will be notified of changes within 72 hours. It is currently anticipated that ESAT or a contracted CLP Laboratory will be used for sample analysis during this project. Laboratory instrumentation and method requirements for the ESAT laboratory are included in Table A.8-3.

B.5 Quality Control

Tables B.5-1 and B.5-2 provide acceptable laboratory QC criteria for ESAT and EPA Region 8. The sample selection for laboratory QC will be determined by the laboratory staff. Where a specific QC criteria table is not provided, the method's QC requirements are met or exceeded by ESAT's and EPA's analytical process.

The calibration procedures for the field measurements to be performed using the fixed-post XRF analyzer are detailed in ESAT SOP# FLD-13.00 *Analytical Determination of Trace Metals in Soil and Sediment by Field Portable X-Ray Fluorescence Spectrometry* (ESAT, 2012). If other multi-probes are used for this sampling event, the field sampling

team will calibrate the probe according to the manufacturer's specifications listed in the owner's manual. The SOPs and procedures appended to this document also detail the associated QA and QC criteria for the field analyses and equipment.

Field QC samples will be collected on the following basis:

- Twenty percent of XRF scanned samples will be shipped to the ESAT Region 8 laboratory as confirmation samples
- Additional collaborative samples will be analyzed at key decision points when the XRF results are close to action levels or when the reliability of the XRF unit is in question; these key points will be determined by the field team leader during field activities
- At least three background samples will be collected and sent to the ESAT Region 8 laboratory for analysis
- During the preliminary sampling event, overall XRF performance will be evaluated by collecting ten soil samples that will subsequently be analyzed by XRF as well as in the laboratory.

B.6 Instrument/Equipment Testing, Inspection and Maintenance

The following chart includes the equipment that will be used during execution of this SAP that requires testing, inspection or maintenance.

Equipment/Instrument	Requirement	Schedule
X-Ray Fluorescence spectrophotometer	Calibration, routine maintenance, scheduled service	In accordance with manufacturer's specifications and user's manual; minimum once daily
Trimble® GeoXT™ GPS	Service	As needed depending on equipment performance
Laboratory analytical instrumentation	Calibration, routine maintenance, scheduled service	In accordance with manufacturer's specifications, user's manual and applicable SOPs

Maintenance and servicing schedules as well as applicable testing criteria are included in the applicable user's manuals as well as SOPs (appended to this document). Note that most spare parts for each piece of equipment are kept at the ESAT Region 8 Laboratory, including parts for field equipment and laboratory instrumentation. Spare parts are routinely available and are ordered during periodic maintenance activities to ensure they are on hand when needed. Service agreements are in place for all laboratory instrumentation to address equipment maintenance, service, parts and repair needs as they arise. Equipment and instrument calibration requirements and frequencies are detailed in the applicable SOPs and user's manuals (attached to this document).

Field equipment will be inspected, tested, and routine maintenance performed prior to deployment in the field by contractor staff members knowledgeable of equipment operation and maintenance requirements at the ESAT Region 8 Laboratory. Any equipment deficiencies and or maintenance requirements will be identified and mitigated (i.e., parts replaced, alternate equipment deployed, etc.). After mitigation, equipment will be re-inspected and the effectiveness of any repairs will be verified. Any repair or maintenance activities performed will be documented in the applicable equipment or instrument log book. Backup equipment will be deployed during these events in case of equipment or instrument failure in the field.

B.7 Instrument/Equipment Calibration and Frequency

As indicated in Section B.6, some laboratory instrumentation (analytical instrumentation) and field equipment (such as the XRF) will require periodic calibration to verify function. Calibration requirements, procedures, testing criteria and deficiency resolution procedures are included in the applicable SOPs and user's manuals, each of which is included in Appendix A of this document (for field equipment). The SOPs and user's manuals for laboratory analytical instrumentation are on file and readily available at the ESAT Region 8 Laboratory. Any variations or inability to calibrate a piece of equipment or instrument will be noted in the relevant logbook, and appropriate mitigation procedures will be followed, or replacement equipment will be obtained. Recalibration of any instrument that requires mitigation of a deficiency will be performed prior to use or deployment.

B.8 Inspection/Acceptance of Supplies and Consumables

All supplies for this event will be purchased by EPA from approved vendors and stored in the field sampling room (or adjacent storage rooms at the ESAT Region 8 Laboratory). The week prior to the sampling event, an EPA or ESAT sampling team member will gather needed supplies and consumables, which will subsequently be verified by an ESAT team member. Supplies and consumables will be ordered, inspected upon receipt, accepted, tracked, and inventoried by the ESAT field biologist at the EPA Region 8 Laboratory. Acceptance of supplies and consumables will be based on the requirements of the end user.

B.9 Use of Existing Data (Non-Direct Measurements)

Non-direct measurements were relied upon for preparation of project implementation. These measurements include previous sampling and analysis plans (EPA, 2009-2013) and historical data provided by ARSG.

B.10 Data Management

Specific management processes will be followed for data likely to be collected during field activities: field equipment calibration and maintenance entries, field logbook entries, chain-of-custody forms, electronically entered and logged data (such as GPS locations, flow measurements, etc.), and analytical data.

Field equipment calibration and maintenance logs – All field equipment calibration and maintenance activities will be documented in a logbook dedicated to each piece of equipment. Logbook entries will be signed and dated by the individual performing calibration or maintenance, or the individual responsible for coordination (such as the field task lead) if equipment is shipped to a manufacturer for repair or maintenance. Field logbooks will be stored with the appropriate piece of equipment. When new logbooks are needed, the former logbook will be stored at the ESAT Region 8 EPA Laboratory, Suite A127 until relinquished to EPA in accordance with ESAT Region 8 contract requirements.

Field logbook/datasheet entries – All field measurements and observations will be recorded in a bound notebook or on appropriate data sheets by the field personnel at the time they are performed. The personnel doing the recording will initial and date each logbook. Corrections to logbook entries will be made by drawing a single line through the error accompanied by the date and the initials of the person performing the correction, followed by the proper entry. Upon return to the ESAT Region 8 laboratory, all data hand entered into field notebooks or datasheets will be transferred to electronic spreadsheets (such as Microsoft® Excel) by ESAT contract staff to prepare for uploading to a SCRIBE project (see below). ESAT field personnel will perform a 100% verification of spreadsheet entries against hand-entered field logbook and datasheet entries before uploading to SCRIBE. Original field notebooks and data sheets will be stored at the ESAT Region 8 EPA Laboratory, Suite A127 until relinquished to EPA in accordance with ESAT Region 8 contract requirements. Non-SCRIBE electronic files generated as a part of this process (i.e., spreadsheets, photographs, scanned logbooks) will be stored on the ESAT Region 8 contractor G drive.

Chain-of-custody forms – When possible, chain-of-custody forms will be generated prior to field activities using SCRIBE and will be filled out when samples are collected following the protocol outlined in *Sample Custody and Labeling* SOP# FLD-11.00 (ESAT, 2012). Otherwise, blank chain-of-custody forms will be used to collect sample information during field activities. Information entered on the forms during investigation activities will be entered into SCRIBE after returning to the ESAT Region 8 Laboratory as a part of the SCRIBE upload process (see below). ESAT personnel will verify 100%

of all the data entered into SCRIBE against the chain-of-custody forms completed in the field. Hard copies of these forms will be stored at the ESAT Region 8 Laboratory, Suite A127 until relinquished to EPA in accordance with ESAT Region 8 contract requirements.

Electronically entered or logged data – In some cases data may be recorded in the field directly on electronic field forms or using data loggers (such as GPS instrumentation, multi-probe data loggers or XRF output). In these cases, upon return to the ESAT Region 8 Laboratory, all electronic data logs will be downloaded directly to a spreadsheet (or alternate electronic media depending on specific instrument software requirements), verified against any hand-written documentation (such as field logs or field data sheets), and processed into an electronic form that can be uploaded directly to SCRIBE. Similarly, electronic field forms will be processed to allow for upload to SCRIBE. Electronic field forms and data logs will be maintained on the ESAT Region 8 contractor G drive. In cases where information must be manually entered into SCRIBE, ESAT personnel will perform 100% verification between electronic documents and data logs and data manually entered into SCRIBE.

Analytical Data – An analytical chemist will log all the samples into LIMS upon receipt at the ESAT Region 8 Laboratory. All analytical results will be uploaded into the LIMS in accordance with SOP# LAB-05.02, *Sample Receipt, Custody, Storage and LIMS Entry of Samples* (ESAT, 2009). Peer review of the data package, at a 100% frequency of reported versus raw data, will be performed by the analytical laboratory before a final report is released. The final report will be in a standard CLP format, including all laboratory and instrument QC results. The laboratory Electronic Data Deliverable (EDD) will immediately be uploaded into a SCRIBE project for permanent electronic storage and archiving after the final report is generated. Hard copies of data reports (including bench sheets) will be stored at the ESAT Region 8 Laboratory, Suite A127 until relinquished to EPA in accordance with ESAT Region 8 contract requirements.

SCRIBE project generation – As indicated above, all data generated as a part of field investigation activities will be uploaded into a SCRIBE project (or update to a SCRIBE project) and subsequently published to Scribe.net in accordance with the *Data Management for Field Operations and Analytical Support,* SOP# 16-DAT-01.00 (ESAT, 2014). It is anticipated that more data may be collected in the field that supersedes existing or historical data that has already been published (such as GPS locations, etc.) for a specific site. Therefore, before data are published or updated to SCRIBE projects, ESAT personnel will perform a 100% verification of each SCRIBE project against data collected in the field (hand entered logbook data, electronic forms and data logs) prior to publishing the project on Scribe.net. Verified SCRIBE projects will be published within

one week of delivery of analytical EDD when possible. The EPA project manager will be immediately notified and an alternate publication date will be established. In the event that conditions preclude publication within that time period, the Task Order Project Officer (TOPO) will be notified and a new publication date will be established.

C. ASSESSMENT AND OVERSIGHT

C.1 Assessment and Response Actions

C.1.1 Field Sampling Assessments

Assessment and oversight of field sampling activities and implementation of the SAP/QAPP will include the following:

- Oversight of field sampling activities
- Oversight of sample handling and chain of custody procedures

The following individuals or their designees are authorized to perform any of the assessments listed above:

- EPA TOPO – Nicole Plescia
- EPA RPM – Paula Schmittdiel
- EPA SAM – Victor Ketellapper
- EPA Toxicologist – Susan Griffin

Assessment of field activities may occur at any time and without prior notice. Only authorized individuals may conduct the assessments and it is their role to issue any corrective action or response action to the situation. If minor problems are identified they will be addressed on-site prior to resuming work. If more significant problems are identified then a stop work order can be issued by the TOPO until the project manager or designee can resolve the problem.

C.1.2 Laboratory Assessments

System assessments of the designated laboratory may be performed by ESAT. The quality assurance officer (QAO), or a designee, may perform a laboratory inspection.

Routine assessments will be conducted at least once a year, in accordance with ESAT's QMP. However, the frequency of the laboratory system assessments will also be based on the level of use and performance of individual designated laboratories. A member of the ESAT team will perform the assessment in accordance with the assessment checklist and TechLaw SOP 02-06-05. The checklist requires examining the laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody

procedures, sample preparation and analysis, instrument operating records, etc. Routine assessments will also be performed before a laboratory is added to the approved laboratory list. Should one-time specialty analysis be requested, the need for on-site assessments will be evaluated and discussed with EPA before an audit.

Performance assessments will require preparing blind QC samples and submitting them along with project samples to the laboratory for analysis. The analytical results of the QC sample analyses are evaluated by the QAO to ensure that the laboratory maintains acceptable QC performance. Performance assessments may be requested by ESAT or EPA. Performance Evaluation (PE) samples will be prepared by and obtained from vendors. The QAO will determine whether a PE sample shall be submitted. PE samples should be submitted if a laboratory has not recently passed an outside PE sample or as requested by EPA.

Response Actions

Corrective Action (CA) may be required at two phases corresponding to the two activities of data generation: 1) field activities (data gathering phase); and 2) laboratory activities (data analysis phase). CAs required as a result of the data analysis phase are initiated by the TechLaw QAO when analytical data are found to be outside the limits of acceptability, as specified in the laboratory SOPs.

C.1.3 Field Corrective Actions

CAs required as a result of the field data collection phase are initiated by the TechLaw field team leader and may result from log reports or field assessments. QC needs to be implemented both during the development of the SAP and sampling activities to ensure that CAs will not be required. CAs are initiated by ESAT if weaknesses or problems are uncovered as a result of field activities. The CAs will depend on the nature or severity of the problem and the level at which the problem is detected, and may include, but shall not be limited to:

- Modifications to sampling procedures
- Recalibration (or replacement) of field instruments
- Additional training of field personnel
- Reassignment of staff personnel
- Re-sampling

C.2 Reports to Management

The results of all laboratory assessments will be submitted to the appropriate ESAT project manager, task order manager, and laboratory assistance team, as well as the EPA Contracting Officer Representative and EPA QA personnel, if requested. An external

assessment of the designated laboratory may also be conducted by EPA at the Region's discretion.

D. DATA VALIDATION AND USABILITY

D.1 Data Review, Verification, and Validation

Laboratory data validation and verification will begin at the sample log-in stage where a sample log-in technician or chemist will compare received samples against chain-of-custody forms and document sample conditions (damage, temperature, etc.). Validation and verification of data will be performed by QA/QC personnel following *EPA National Functional Guidance for Inorganic Data*, (EPA, 2002) in order to determine if the DQOs were met. Sample data deemed outside the expected range will be investigated, communicated to the analytical chemistry staff, flagged (if needed) and potentially re-sampled to verify or discredit the data. Data that have proven to be incorrect may be flagged, further reviewed, or invalidated. The cause of incorrect data will be investigated and appropriate response actions will be taken, including communication of any issues to the user in the data report. All XRF data will only be verified, as opposed to validated.

Uncertainty of validated data will be evaluated by the RPM and SAM to determine if the DQOs were met. In the event that the DQOs were not met, they will be reviewed to determine if they are achievable and may be revised if necessary, and the data may be further evaluated to determine the impact to the project. Data usability and limitations will be evaluated by the RPM and SAM.

Abbreviated verification will be completed on ten percent of the analytical results for data that is electronically uploaded directly from the analytical instrumentation into the ESAT LIMS. This will be performed to ensure that data were produced in accordance with procedures outlined in this project plan. The following elements will be reviewed for compliance as part of the abbreviated data validation:

- Holding Times
- Calibration
- Blanks
- Spikes
- Duplicates
- LCSs
- Reporting Limits
- Analyte Quantification

D.2 Verification and Validation Methods

The analytical data will be validated for ten percent of the results by either the acting EPA ESAT Region 8 Laboratory QAO or by a designated TechLaw, Inc. QAO outside of the Region 8 ESAT office. The validation will include reviewing ten percent of the samples for 100% of the analytical analysis performed and reported. The following elements will be reviewed for compliance as part of the abbreviated data validation:

1. Holding Times
2. Calibration
3. Blanks
4. Spikes
5. Duplicates
6. LCSs
7. MSs
8. Post Digest Spike (optional)
9. Internal Control Standard
10. Dilution Sample
11. Reporting Limits
12. Analyte Identification
13. Analyte Quantification
14. Comparison of hardcopy results to EDD

Data validation will conform to the EPA Contract Laboratory Program National Functional Guidelines for inorganic data and will use standard data qualifiers as described below.

D.3 Reconciliation with User Requirements

The following definitions provide brief explanations of the national qualifiers assigned to results in the data review process. If the regions choose to use additional qualifiers, a complete explanation of those qualifiers should accompany the data review.

U	The analyte was analyzed for, but was not detected above the level of the reported sample Quantitation limit.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the results may be biased high.
J-	The result is an estimated quantity, but the results may be biased low.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be presented in the sample.

UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise

D.4 Reconciliation with DQOs

Information obtained from the field investigation will be evaluated through the Data Quality Assessment (DQA) process to determine if the data obtained are of adequate quality and quantity to support their intended use. The DQA process consists of five steps, as summarized below (EPA, 2006):

1.) *Review the project's objectives and sampling design*: Review the objectives defined during the systematic planning to assure that they are still applicable. If objectives have not been deployed, specify them before evaluating the data for the projects objectives. Review the sampling design and data collection documentation for consistency with the project objectives observing any potential discrepancies.

2.) *Conduct a preliminary data review*: Review QA reports (when possible) for the validation of data, calculate basic statistics, and generate graphs of the data. Use this information to learn about the structures of the data and identify patterns, relationships, or potential anomalies.

3.) *Select the statistical method*: Select the appropriate procedures for summarizing and analyzing the data based on the review of the performance and acceptance criteria associated with the project objectives, the sampling design, and the preliminary data review. Identify the key underlying assumptions associated with the statistical tests.

4.) *Verify the assumptions of the statistical method*: Evaluate whether the underlying assumptions hold, or whether departures are acceptable given the actual data and other information about the study.

5.) *Draw conclusion from the data*: Perform the calculations necessary to draw reasonable conclusions from the data. If the design is to be used again, evaluate the performance of the sampling design.

Uncertainty of validated data will be evaluated by the RPM and SAM to determine if the DQOs were met. In the event that the DQOs were not met, they will be reviewed to determine if they are achievable and may be revised if necessary, and the data may be further evaluated to determine the impact to the project. Data usability and limitations will be evaluated by the RPM and SAM.

References:

U.S Environmental Protection Agency (EPA), 1994. "*Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry*", Method 200.7 Revision 4.4, May 1994.

U.S Environmental Protection Agency (EPA). 1998b EPA "*Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*", SW-846, Method 7473, Revision 0, January 1998.

U.S. Environmental Protection Agency (EPA), 2000. "*Guidance for the Data Quality Objectives Process*." EPA QA/G-4. Copies may be obtained from the Quality Staff Home Page: www.epa.gov/quality August 2000.

U.S. Environmental Protection Agency (EPA), 2001a. "*EPA Requirements for Quality Assurance Project Plans*" EPA QA/R-5 March 2001.

U.S. Environmental Protection Agency (EPA), 2001b. EPA QA/G-5i 2001. Guidance on Data Quality.

U.S Environmental Protection Agency (EPA), 2001c. *Requirements for Quality Assurance Project Plans*" (EPA QA/R-5").

U.S. Environmental Protection Agency (EPA), 2002. "*Choosing a Sampling Design for Environmental Data Collection*" (EPA QA/G-5S) December 2002.

U.S. Environmental Protection Agency (EPA), 2002. "*Guidance for Quality Assurance Project Plans*" (EPA QA/G-5).

U.S. Environmental Protection Agency (EPA), 2003. "*Superfund Lead-Contaminated Residential Sites Handbook*." (OSWER 9285.7-50).

U.S. Environmental Protection Agency (EPA), 2006. "*Guidance on Systematic Planning Using the Data Quality Objectives Process*" (EPA QA/G-4).

U.S. Environmental Protection Agency (EPA), 2007. "*Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment*", Method 6200, Revision 0, February 2007.

U. S. Environmental Protection Agency (EPA), 2011. *Contract Laboratory Program Guidance for Field Samplers.*

Standard Operating Procedures:

Environmental Services Assistance Team (2014) *Data Management for Field Operations and Analytical Support.*SOP# 16-DAT-01.00

Environmental Services Assistance Team (2012) *Soil Sampling*. SOP# FLD-05

Environmental Services Assistance Team (2012) *Global Positioning System (GPS) – Trimble GeoXT 2008 series.* SOP# FLD-07

Environmental Services Assistance Team (2012) *Sample Custody and Labeling*. SOP# FLD-11

Environmental Services Assistance Team (2012) *General Field Sampling Protocols*. SOP# FLD-12

Environmental Services Assistance Team (2012) *Analytical Determination of Trace Metals in Soil and Sediment by Field Portable X-Ray Fluorescence Spectrometry.* SOP# FLD-13

Environmental Services Assistance Team (2007) *Collection, Analysis, and Disposal of ESAT Laboratory Waste*. SOP# LAB 01.01

Environmental Services Assistance Team (2009) *Sample Receipt, Custody, Storage and LIMS Entry of Samples* SOP# LAB 05.02

TechLaw (2012) *Field Procedures - Analytical Support and Laboratory Selection*. SOP 02-06-05

Tables

Table A.7-T SRLOC Contaminants of Concern, Detection Limits and Methods

Target Analytes	EPA Digestion Method[1]	EPA Method[1]	Instrument	Fraction Evaluated	Sample Volume	Preservation	Holding Time	Laboratory PQL (mg/kg)	Sediment Screening Benchmark (mg/kg)
Aluminum (Al)	200.2	200.7	ICP-OE	TR	200 grams	Ice	180 days	5	25,519
Beryllium (Be)	200.2	200.7	ICP-OE	TR				0.5	NA
Calcium (Ca)	200.2	200.7	ICP-OE	TR				25	NA
Chromium (Cr)	200.2	200.7	ICP-OE	TR				0.5	43
Copper (Cu)	200.2	200.7	ICP-OE	TR				1	32
Iron (Fe)	200.2	200.7	ICP-OE	TR				25	188,400
Magnesium (Mg)	200.2	200.7	ICP-OE	TR				25	NA
Manganese (Mn)	200.2	200.7	ICP-OE	TR				0.5	631
Zinc (Zn)	200.2	200.7	ICP-OE	TR				2	121
Antimony (Sb)	200.2	200.8	ICP-MS	TR				0.1	2
Arsenic (As)	200.2	200.8	ICP-MS	TR				0.2	9.8
Cadmium (Cd)	200.2	200.8	ICP-MS	TR				0.02	1
Lead (Pb)	200.2	200.8	ICP-MS	TR				0.02	36
Nickel (Ni)	200.2	200.8	ICP-MS	TR				0.1	23
Selenium (Se)	200.2	200.8	ICP-MS	TR				0.1	NA
Silver (Ag)	200.2	200.8	ICP-MS	TR				0.05	1
Thallium (Tl)	200.2	200.8	ICP-MS	TR				0.1	NA
Mercury (Hg)	7473	7473	CVAA	Total			28 days	0.02	0.18

Notes:

TR - Total recoverable metals from soil

PQL - Practical Quantitation Limit

mg/kg - milligrams per kilogram

Sample containers should be plastic and capable of holding the required sample volume.

[1]EPA's *Methods for the Determination of Metals in Environmental Samples*, Supplement I, May 1994 (Series 200 Methods)

[2]*Development and Evaluation of consensus-Based Sediment Quality Guidelines for Freshwater Ecosystems.* Environ. Contam. Toxicol. Vol. 39, pp. 20-31. 2000.

[3]Not applicable - no established criteria for aluminum

[4]*Consensus-based sediment guidelines: Recommendations for use & application*. Wisconsin Dept. of Natural Resources, Madison, WI. Report No. WT-732-2003. December, 2003.

Table A. L-4Û Sampling Locations and Activities -2014

Station Location	Site Description	Composite Surface Soil[1] Total Recoverable Metals	Biased High Discrete Surface Soil [1] Total Recoverable Metals	Subsurface Soil Total Recoverable Metals
PA-SPS	Silverton Public Schools	1	10	1
PA-SFLC	Silverton Family Learning Center	1		1
PA-SMP	Silverton Memorial Park	1		1
PA-SPL	Silverton Public Library	1		1
PA-SPO	Silverton Post Office	1		1
PA-TH	Town Hall	1		1
PA-KMRC	Kendall Mountain Recreation Area	1		1
PA-CP	Columbine Park	1		1
PA-SJPLC	San Juan Public Lands Center	1		1
PA-SMHC	Silverton Mining Heritage Center	1		1
R001 through R075	50 to 75 residences to be determined in the field based on access	1 to 4 per residence depending on size		1 per residence
BG001	Background sample	1		
BG002	Background sample	1		
BG003	Background sample	1		
SS001	Source or Slag sample	1		
SS002	Source or Slag sample	1		
SS003	Source or Slag sample	1		
SS004	Source or Slag sample	1		
SS005	Source or Slag sample	1		
SS006	Source or Slag sample	1		

1 A total of 10-12 of the samples will be evaluated for in-vitro bioaccessibility

Table B.1-1 Sampling Checklist

1. Make sure the necessary paperwork is in place for a field event: Approved LSR, SAP, and QAPP.

2. Coordinate sampling dates and times with members of the field team and talk with chemists involved in the project to see if your plans work for them. Coordinate sample delivery with outside laboratories.

3. Fill out the necessary paperwork: Comp Time forms and TAs if travel will be more than 50 miles from the laboratory. Be sure to have reservations made for airlines and hotels if necessary.

4. Make necessary arrangements with people outside of the Region VIII laboratory that are involved with the project. Arrange meeting times and places, vehicle needs, sampling teams, additional equipment needs, etc.

5. Inform any volunteers outside of the EPA laboratory group what will be involved with sampling - physical stressors, equipment to bring, lunch, water, etc.

6. Calibrate XRF spectrometers needed for fieldwork well-before leaving.

7. Lay out needed sampling equipment in the field room (see attached list).

8. Check vehicles: fill with gas, top off windshield wiper fluid, equip with cell phones, walkie-talkies, and chargers.

9. Charge batteries for needed sampling equipment one or two nights before leaving: digital camera, hydrolab, GPS units, walkie-talkies, etc.

10. Pack vehicles the night before leaving. In the event of hot or cold weather, leave meters and deionized water in the field room and pack the day you leave.

11. In the event of a day-trip, calibrate meters the morning you leave.

Table B.1-2 Field Equipment List

Sample Containers:
4oz or 8oz glass sample jars

Soil Sampling:
Plastic disposable scoops
Sieves
Drying oven
Brushes
Stainless steel bowls
Stainless steel spoons
Stainless steel shovel
Plastic baggies
Alconox decon rinse
HNO_3 decon rinse
DI water
Tarp
Paper towels
Sharpies
Spare gallons of DI H_2O
Flag markers
GPS unit (charged)

XRF Analysis:
XRF gun
Spare batteries (charged)
XRF standards
XRF control unit
Tripod stand
XRF cups
Mylar film

Paperwork:
SAP/HASP
Maps/Gazetteer
Chains
Labels
Field notebook(s)
Pens
Markers

Misc:
Battery charger
pH test strips
Nitrile gloves
Neoprene gloves
Safety glasses
Kimwipes
Trash bags
Plastic bags
Tape
Coolers
DI rinse bottles
Cell phones w/ charger
Spare car keys
Vehicle log
E-pure water
DI water
Compass
Tape measure

Summer Field Gear:
Backpacks
Hiking boots
Hat
Rain gear
Wool socks
Layered clothing
Sunscreen chapstick
Bug spray
Sun glasses
Water/food
Pocket knife

Table B.5-1
ESAT Region 8 - Metals QC Criteria

QC Check / Symbol	Explanation	Run Frequency	Acceptance Criteria	Corrective Action
Initial Calibration Verification (ICV)	Certified standard or standard from a different lot/source than calibration standards	Beginning of run to verify calibration	90-110% recovery (%R) of "true value"	Terminate analysis, restandardize
Continuing Calibration Verification (CCV)	Approximate mid-range standard made from working standards stock	Every 10 unknowns and at end of run	90-110%R "True" value	Re-analyze immediately (once). Then: Restandardize and rerun all samples following last "acceptable" CCV. If recovery >110% and <120% and all associated samples (same analyte) show non-detected, no action required.
Spectral/Mass Interference Check for ICP-OE & ICP-MS (ICSA / ICSAB)	Analyze spectral interferents at high concentrations alone (ICSA) and with other target analytes (ICSAB) to evaluate the effect on analyte recovery	Once per analytical run, prior to sample analyses	ICSAB: ± 20%R 'true value' ICSA: ± 20%R 'true value' or < ±PQL whichever is greater	Evaluate the sample analyte levels. Rerun ICSA/AB or use an alternate wavelength. If interferent levels in the samples don't approach ICSA interferent levels, no action is required. If necessary, recalculate IECs & rerun associated samples
Calibration Blanks, Initial & Continuing (ICB & CCB)	Blank with same reagents as working standards; i.e. zero point on curve	Beginning, end, and after each ICV/CCV during analytical run	≤ ±PQL	Re-analyze immediately once. If still unacceptable, terminate analysis & restandardize. Rerun all samples analyzed after last "acceptable" blank. Evaluate interferent level(s) vs samples, use prof judgement for addit'l required sample reruns.
Preparation Blank (PB)	Digested or prepared blank processed identical to samples. Aliquot of clean water prepared using same reagents/volumes as unknown samples.	Once per preparation batch/per matrix, or at 5% frequency, whichever is greatest	≤ ±PQL	PB > PQL: Redigest all samples >MDL and <10x PB value PB < -PQL: Re-calibrate and re-analyze all associated samples
Matrix Spike & Matrix Spike Duplicate (MS & MSD)	Unknown sample (NOT a field blank) fortified at approximately 10-100x MDL for each target analyte. High concentration samples (spike <25% sample target analyte concentration), no calculation is required	1 per 20 unknowns per matrix, whichever is greatest (One PB Spike per PB)	Spike recovered at: 80-120% (ICP& MS) - waters 65-135% (all) - solids	Compose 1 post-digest spike (PS) and retest, note in the narrative. (Analyze original sample with PS) Evaluate duplicate reproducibility. Compare results to LFB/PBS for similar trends. If no similar trends observed, assume a matrix effect. Qualify corresponding analyte data as estimated 'J' for similar matrix samples in set.
Lab Fortified Blank (LFB or PBS)	Spike of reagent blank at same level as MS (analyze/prep identical to samples)	Recommend: once/run	85-115%R of expected (for targe analytes)	Used for comparison to Matrix Spike. If MS/MSD in-control no corrective action necessary.
Lab Control Sample (LCS)	For solid & liquid digested samples. A known of similar matrix prepared the same as unknown samples.	1 per prep batch or one per matrix, whichever is greater.	Aq: 80-120%R of "true" Solids: 70-130%R of "true" or published limits	Recalibrate & reanalyze. If still unacceptable, check for corresponding high or low results in pre-digest spikes, if similar, redigest all associated samples
Serial Dilution (L)	Sample analyzed at 5x the reported analysis. (for matrix effect evaluation) Applies to analytes >50x MDL (in the original analyzed solution)	1 per 20 unknown	Diluted value 90-110% of original analysis.	Concentrations compared/reported from the analyzed solution only. Check IECs and re-analyze. May re-analyze both sample and 'L' at a higher dilution. Use professional judgement, and discuss outliers in the narrative.
Detection Limit Standard (CRI/CRA)	Low level standard ≈3-5x MDL concentration. Applies to all target analytes except Al, Ca, Fe, Mg, Na, & K	Once per analytical batch prior to unknowns	50-150%R for Sb, Pb, and Tl. 70-130%R for other target analytes*.	1. Rerun 2. If all associated samples ≥CCV for outlier analyte, no action required 3. Correct instrument's sens. problem or else need to redetermine and raise reporting limits *[Al, Ca, Fe, Mg, Na, & K are monitored without corrective actions]
ICP-MS Internal Standard (IS)	IS standard solution added to all samples, blanks, and standards.	All samples and standards corrected for IS response.	60% - 125%R of IS associated with target analyte(s)	[IS recovery determined versus calibration blank response.] Dilute sample by 2, re-analyze. Continue to dilute until IS %R acceptable.

Table B.5-2
QA/QC Calculation Algorithms

Statistical QC Parameter Evaluated	Acronym	Analyses Applied to	Calculation Algorithm
Percent Recovery	%R	Spike recovery determinations	$\%R = ((C_s - S_a) \div (S_a)) \times 100$
Percent Recovery	%R	ICV/CCV, ICSAB, LCS	$\%R = (A_T \div T) \times 100$
Relative Percent Difference	RPD	Variance between duplicates	$RPD = ((C - C_D) / ((C + C_D) \div 2)) \times 100$
Percent Difference	%D	Serial dilution variance	$\%D = ((C - C_L) / C) \times 100$

Notes:

C = Sample extract concentration

C_s = Sample extract, spiked concentration

S_a = Spike amount added

T = True (possibly certified) amount in standard solution

Hardness = (Ca, mg/L)*2.497 + (Mg, mg/L)*4.118

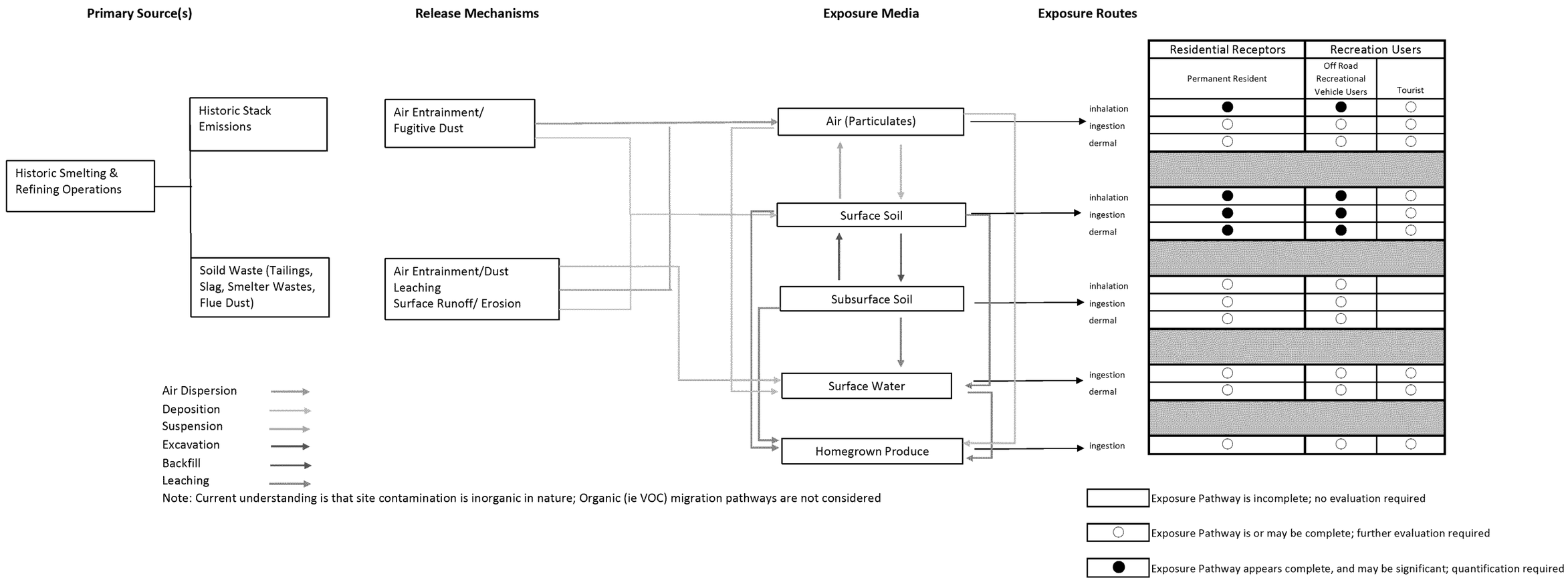
C_D = Duplicate sample concentration

C_L = Sample extract concentration, dilution factor corrected.

AT = Analyzed concentration for the known standard.

Figures

Figure A.7-1 Site Conceptual Exposure Model



Appendix A
Standard Operating Procedures

16-DAT-01.00
Data Management for Field Operations and Analytical Support

Technical Approval: Mark McDaniel

PRINT NAME

SIGNATURE **Date:** 3/12/14

QA Management Approval: Nikki MacDonald

PRINT NAME

SIGNATURE **Date:** 03/12/14

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1.0 SOP Description

The purpose of this standard operating procedure (SOP) is to provide a consistent format for all Region 8 Environmental Services Assistance Team (ESAT) data management personnel who perform uploads to Scribe and management of associated databases and reports.

This SOP is applicable to all ESAT personnel who prepare, process, review, and load analytical data into the Scribe database for the Field Operations Group and the Analytical Support and Data Validation Group.

2.0 Abbreviations and Acronyms

EDD	Electronic Data Deliverable
ERT	Environmental Response Team
ESAT	Environmental Services and Assistance Team
LIMS	Laboratory Information Management System
SOP	Standard Operating Procedure
TDF	Technical Direction Form
TO	Task Order
USEPA	United States Environmental Protection Agency

3.0 Health and Safety

All office-related safety precautions must be followed. Consideration is given to ergonomics for staff members using a keyboard and sitting in front of a computer terminal for extended periods of time and all other work conditions where ergonomics may be an issue.

4.0 Equipment and Supplies

Standard office supplies are required for this SOP, such as a personal computer and central filing system. Specific equipment and supplies are listed below:

- Internet connection and access to the ESAT network drive
- Access to Scribe and the Scribe databases associated with Technical Direction Forms (TDFs) issued by the client
- Login capabilities to the Scribe.NET website
- Microsoft Office software applications
- External hard drive containing the appropriate databases for upload

5.0 General Procedures

ESAT personnel are responsible for acquiring, compiling, reviewing, and loading analytical data into the appropriate Scribe database associated with a specific TDF. Electronic Data Deliverables (EDDs) are posted to the network drive from the Laboratory Information Management System (LIMS) on or before the EDD due date.

5.1 Review, Obtain, and Prepare EDDs to be uploaded to Scribe

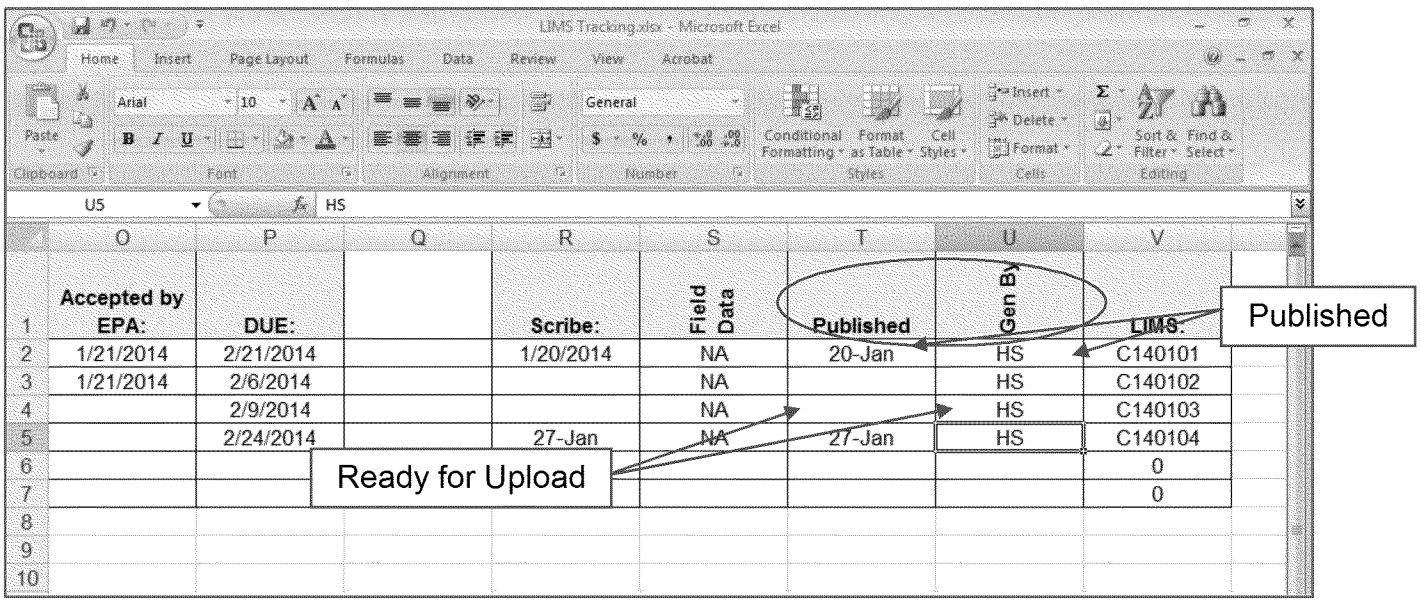
The analysts and/or Data Package Coordinator posts EDDs to the appropriate Task Order (TO) and project folder on the network drive upon completion. Prior to uploading or publishing a project to Scribe, review the LIMS Tracking spreadsheet to ensure that the

Data Package Coordinator has completed assembly and finalization of the current Sample Event(s) (Figure 5.2). EDDs that are complete and ready for upload are listed in the LIMS Tracking spreadsheet (located on the network drive), and will be signed off in the “Gen By” (generated by) column (Figure 5.1). EDDs that have been published will contain a date in the “Published” column (Figure 5.1).

Note: TO Numbers and TDFs are not permanent, and are subject to change based on the contract year, as well as the type and number of sample events.

- Review the "Gen By" and "Published" columns. If the Data Package Coordinator's initials are listed in the "Gen by" column, but a date is not included in the "Published" column, that sampling event is ready for uploading to the database.

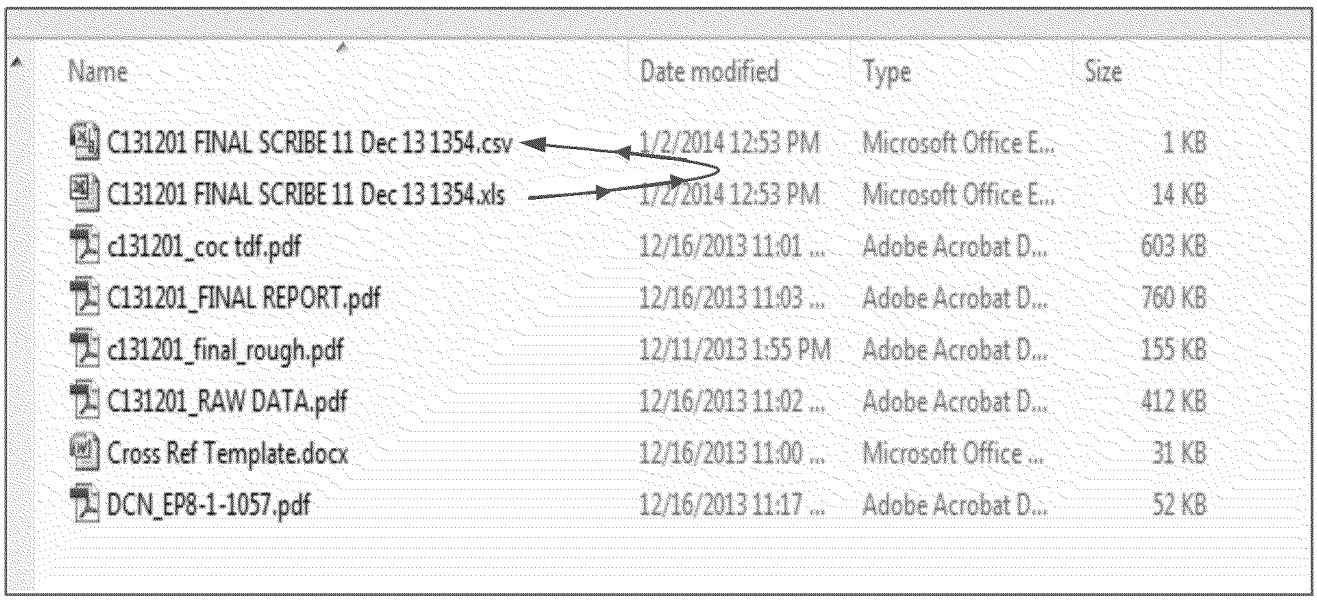
Figure 5.1 LIMS Tracking Spreadsheet Example



	O	P	Q	R	S	T	U	V
1	Accepted by EPA:	DUE:		Scribe:	Field Data	Published	Gen By	LIMS:
2	1/21/2014	2/21/2014		1/20/2014	NA	20-Jan	HS	C140101
3	1/21/2014	2/6/2014			NA		HS	C140102
4		2/9/2014			NA		HS	C140103
5		2/24/2014		27-Jan	NA	27-Jan	HS	C140104
6								0
7								0
8								
9								
10								

- Navigate to the TO and Projects to be uploaded
- Select the .xlsx EDD files, and within the same folder, convert them to .csv format for uploading to Scribe
- Repeat with all other EDDs noted from the LIMS Tracking spreadsheet

Figure 5.2 Example of Sample Event Folder from the Network Drive



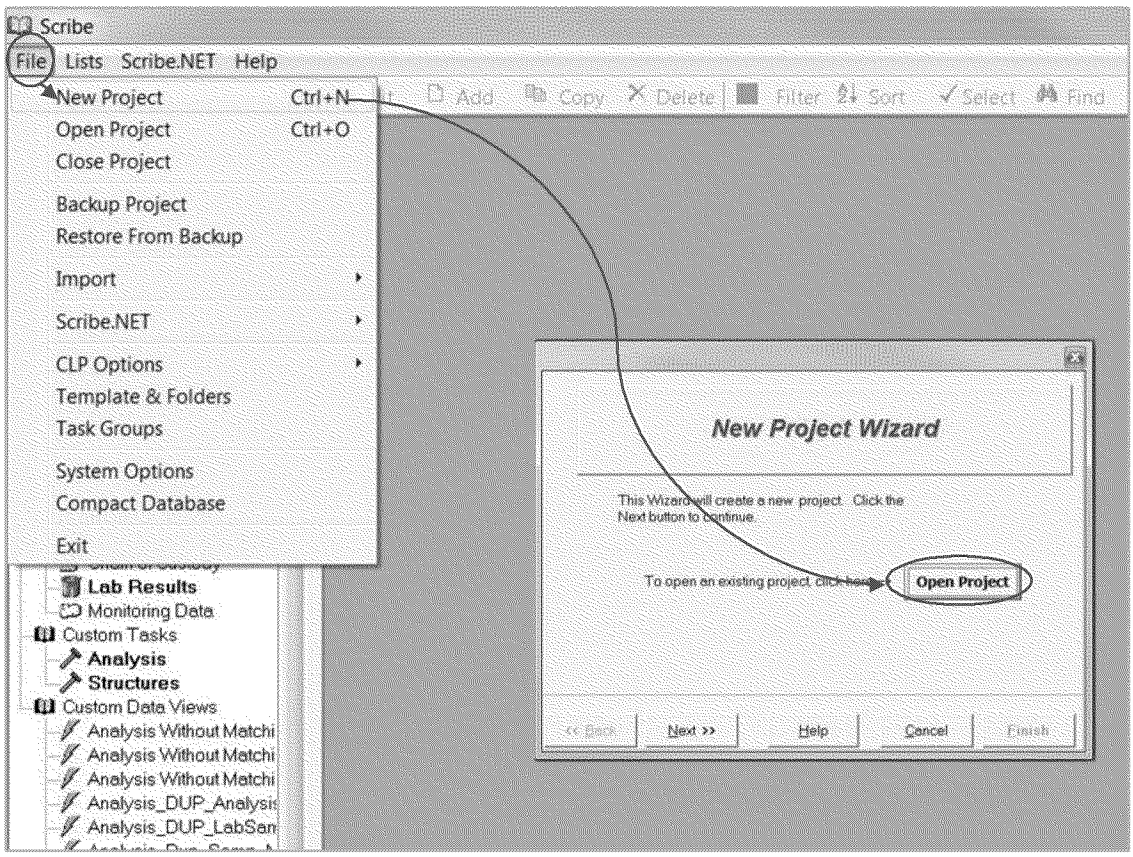
Name	Date modified	Type	Size
C131201 FINAL SCRIBE 11 Dec 13 1354.csv	1/2/2014 12:53 PM	Microsoft Office E...	1 KB
C131201 FINAL SCRIBE 11 Dec 13 1354.xls	1/2/2014 12:53 PM	Microsoft Office E...	14 KB
c131201_coc tdf.pdf	12/16/2013 11:01 ...	Adobe Acrobat D...	603 KB
C131201_FINAL REPORT.pdf	12/16/2013 11:03 ...	Adobe Acrobat D...	760 KB
c131201_final_rough.pdf	12/11/2013 1:55 PM	Adobe Acrobat D...	155 KB
C131201_RAW DATA.pdf	12/16/2013 11:02 ...	Adobe Acrobat D...	412 KB
Cross Ref Template.docx	12/16/2013 11:00 ...	Microsoft Office ...	31 KB
DCN_EP8-1-1057.pdf	12/16/2013 11:17 ...	Adobe Acrobat D...	52 KB

5.2 Scribe Data Load

Each TO project/TDF has its own database. Several separate sample events may occur under each project and will be managed according to the associated TO and project.

- Access the external hard drive containing the appropriate TO databases
- Open the Scribe Database Program
- Select "File" from the menu then "New Project"
- Select "Open Project" in the "New Project Wizard" dialogue box (this will open a Windows Explorer window from which to choose the appropriate folder pathway and database file)

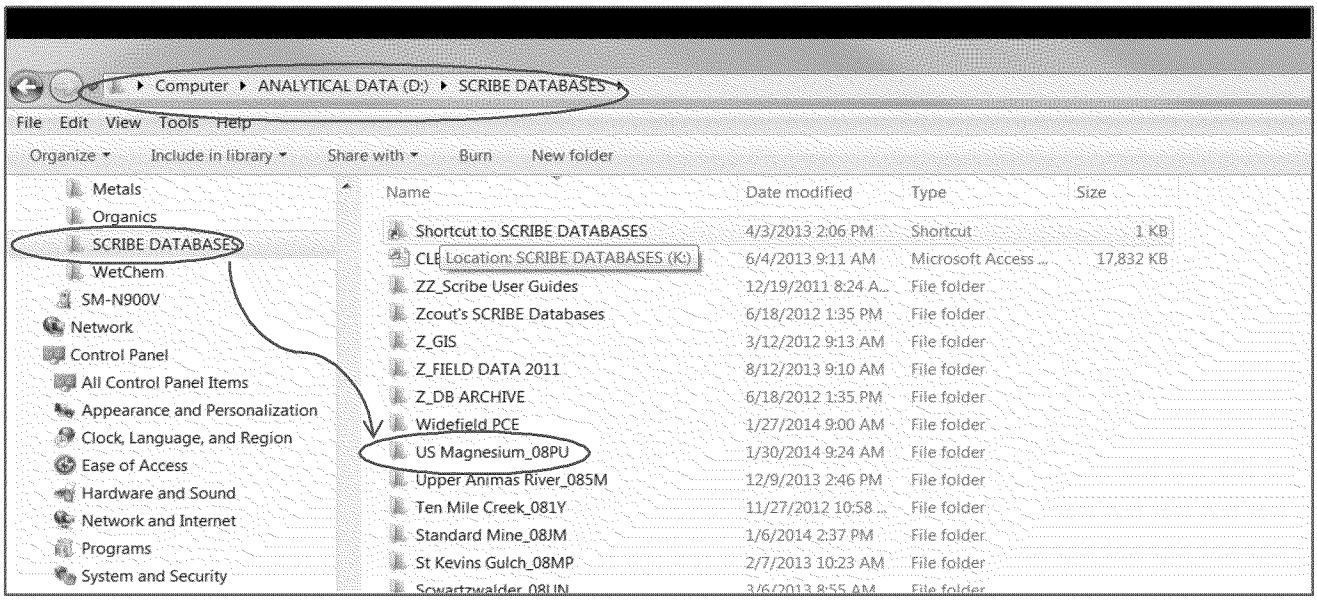
Figure 5.3 Scribe Database "Open Project"



- Navigate to the current TO and Project to be uploaded

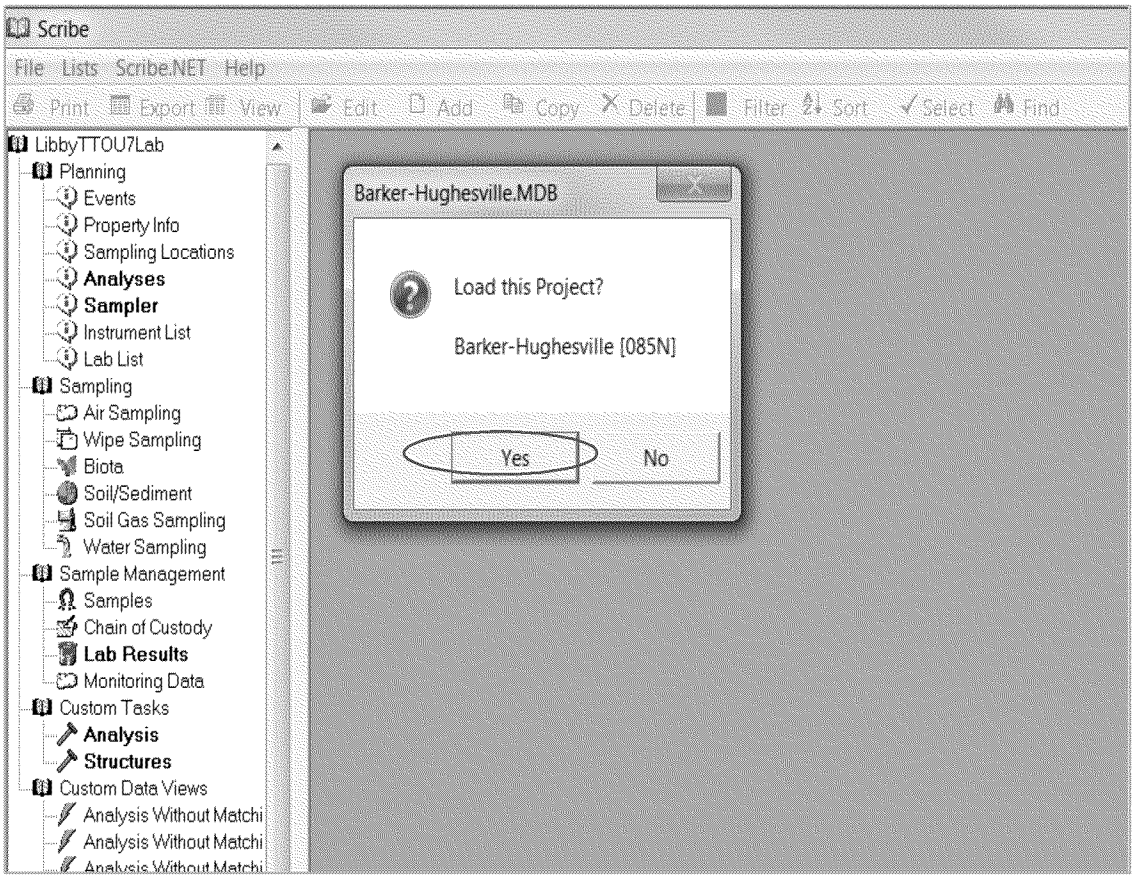
  Note: Databases for the current TO are stored on an external hard drive as shown in Figure 5.4, but the external storage location, pathways, and TO numbers are subject to change.

Figure 5.4 Scribe Database Project Pathway



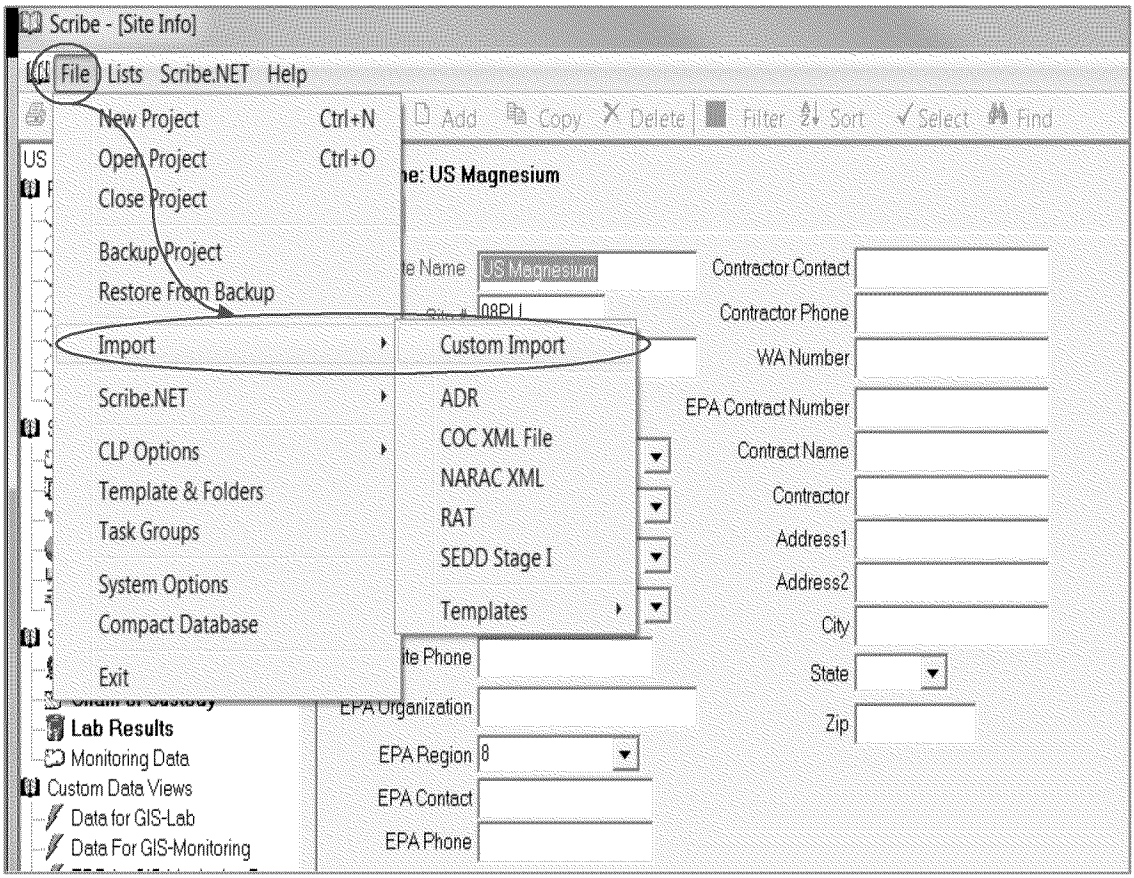
- Select "Yes" in the "Load this Project" dialogue box

Figure 5.5 Scribe Database Load Project



- Select "File -> Import -> Custom Import"
- Select "No" in the resulting "Backup Now?" dialogue box

Figure 5.6 Scribe "File/Import/Custom Import"

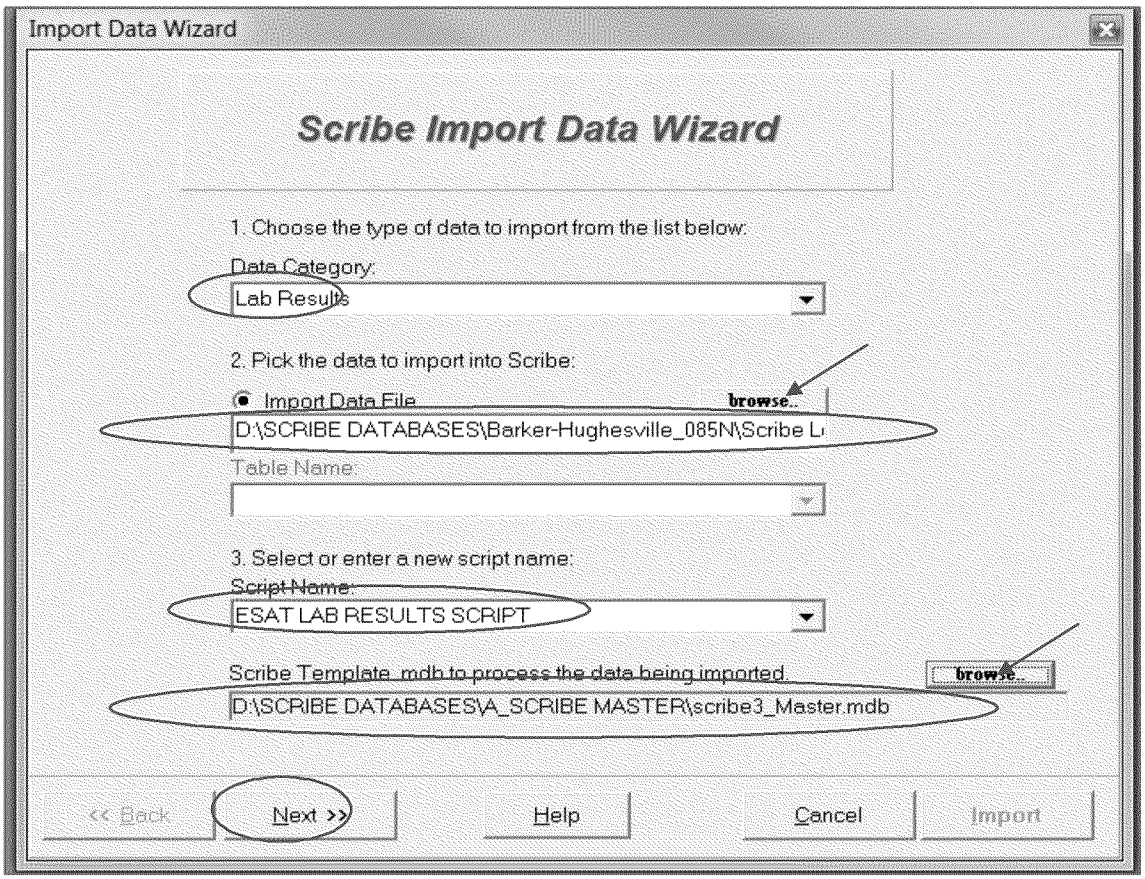


- Select "Lab Results" from the drop down arrow list in the Data Category field
- Select the correct EDD using the "browse" button above the Import Data File field

  Note: EDDs will be obtained from the folders located on the network drive pathways discussed in Section 5.1.

- Select "ESAT Lab Results Script" for the "Script Name" field
- Select the Master Scribe template using the "browse" button above the Scribe Template field; this template is located on the external drive in Scribe Databases/Master
- Select "Next"

Figure 5.7 Scribe "Import Data Wizard"

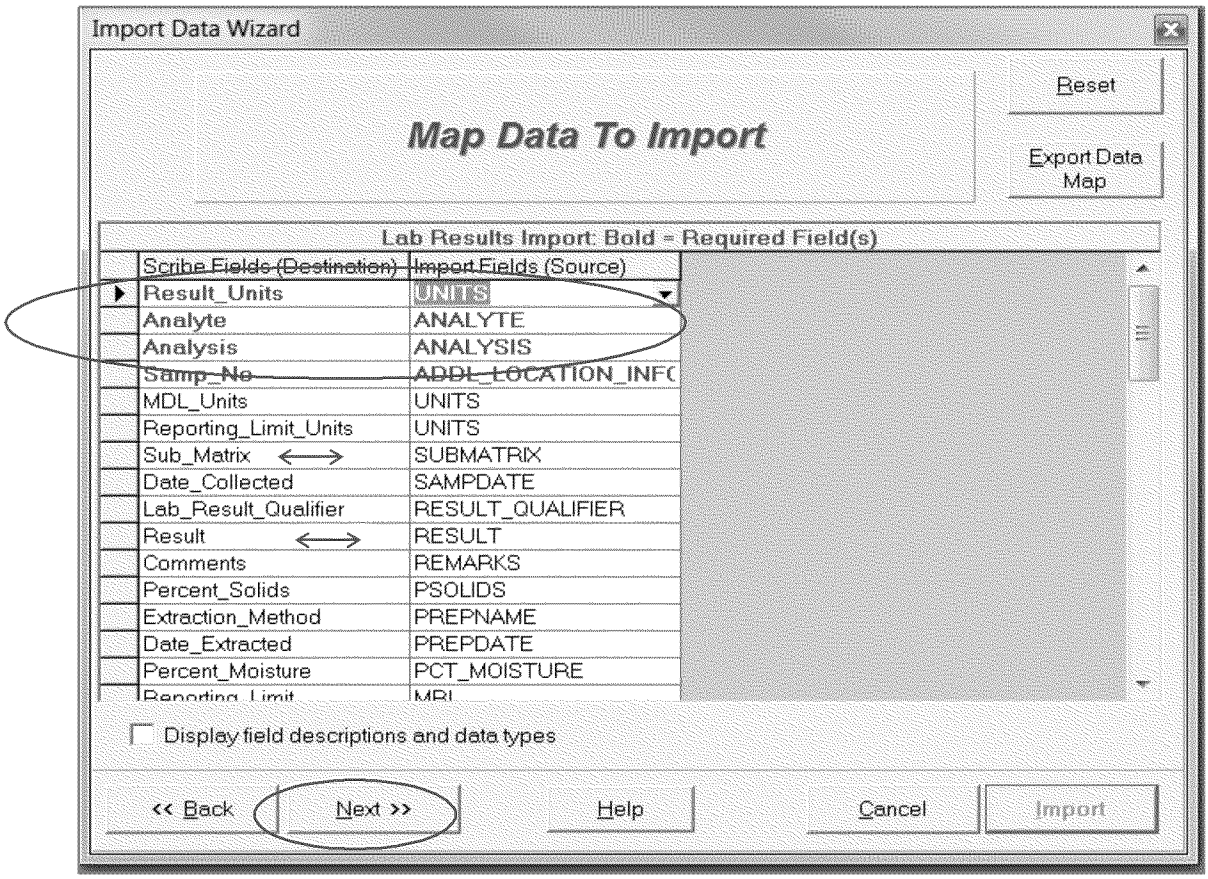


- Review the field mapping dialogue box to ensure that all fields match up with Scribe requirements

  Note: The fields in blue bolded text are required fields. The remaining fields (listed below the blue bolded font) may not match exactly in name. Ensure that they match in type and meaning regardless of the slight differences in names and case. In addition, not all "Import Fields" fields will be present for "Scribe Fields".

- Select "Next"

Figure 5.8 Scribe "Map Data to Import"

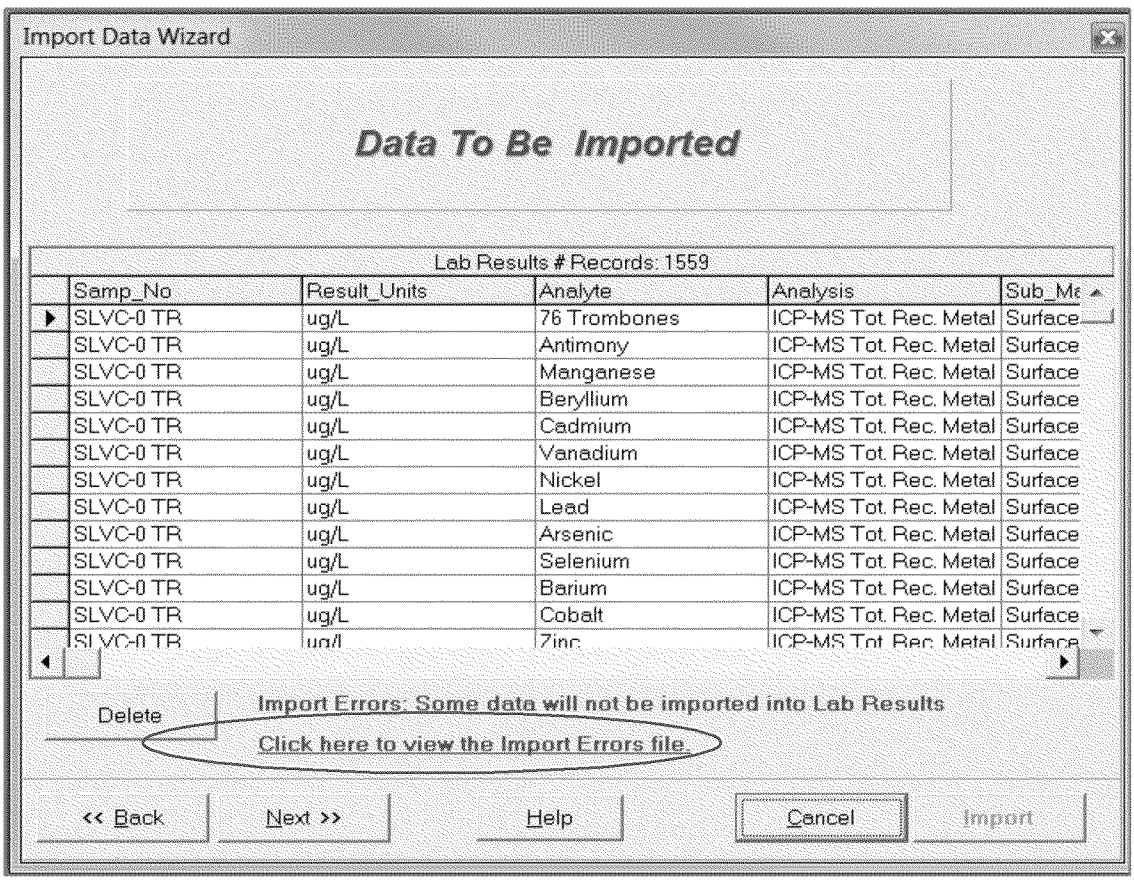


- Review the total in the "Lab Results # Records:" field
- Ensure that the total number of records in the "Lab Results # Records" field matches the total in the EDD

  Note: The Excel formatted EDD can be opened and reviewed to verify that the total number of samples to be imported matches the number of samples contained in the EDD.

- Select the blue bolded "Import Errors file" link to obtain a list of errors if the import is unsuccessful (list will open in Excel format)

Figure 5.9 Scribe "Data to be Imported" Import Errors



- Review the error report, open the appropriate EDD, and correct the errors. As shown in the example report below, the "Reporting Units" field has a numerical value, which is incorrect. Because this is a text field, the necessary correction would be to insert the appropriate unit text, which for this EDD would be ug/L.

  Note: The example below contains one of the common types of errors that may occur. That is, one that is correctable within the EDD itself and by the Scribe uploader. However, this is not the only possible error type. If a more complex error occurs, one that cannot be corrected within the EDD, an ESAT analyst may need to be contacted for assistance and the error may need to be corrected at an earlier point in the process.

  Errors within the Scribe program itself are essentially non-existent for the Analytical Support Group and Field Operations databases. If an upload error occurs, it is generally caused by incorrect selections of either the database script or the Scribe Master template. In those cases, return to the import screen and ensure that all scripts and templates selected are correct. If the database still shows errors that are not correctable in the EDD, the error(s) may be Scribe-related. If an error occurs that cannot be corrected within the EDD, or by correcting possible upload procedure errors, contact the Environmental Response Team (ERT) Software Support department for assistance. ERT can be contacted by email: ertsupport@epa.gov or by phone: 800-999-6990.

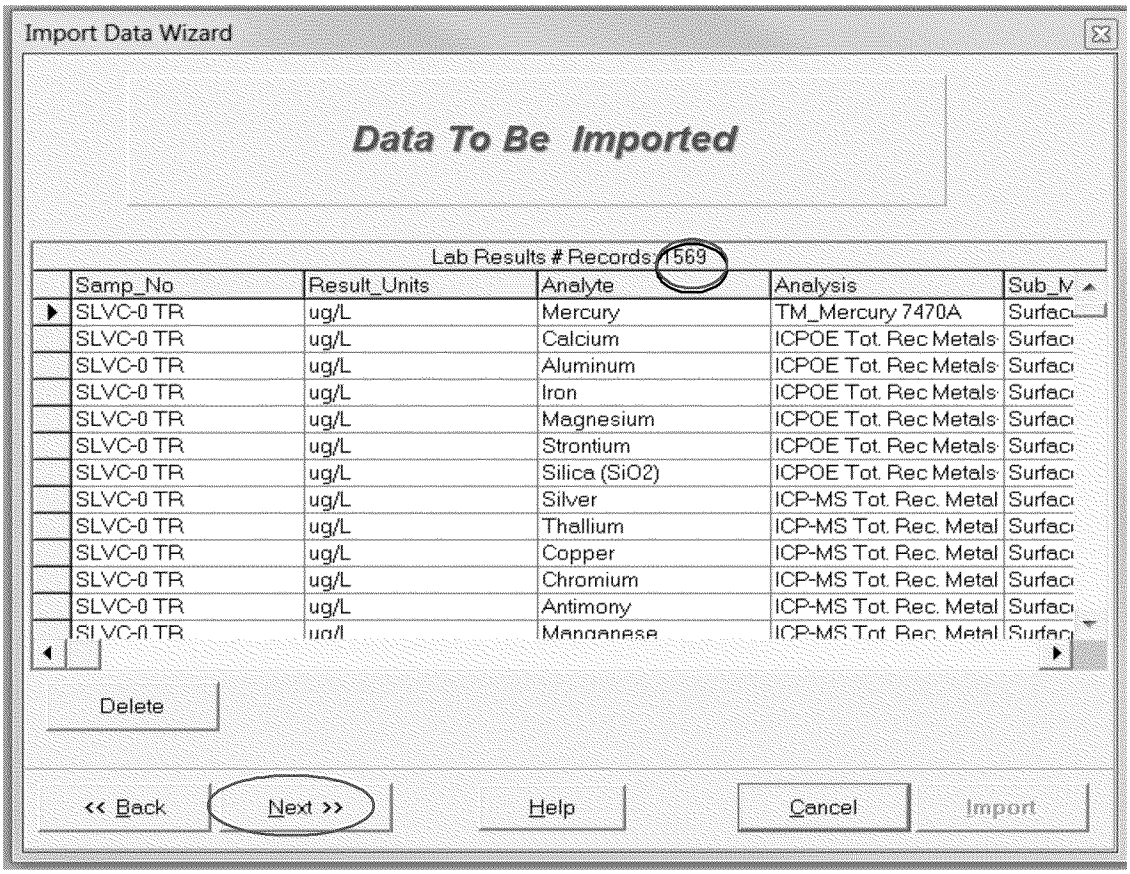
Figure 5.10 Scribe Example of Import Errors File

L2 | Reporting_Limit_Units

	A	B	C	D	E	F	G	H	I	J	K	L	M	N
1	The following data will not be imported for Lab Results:													
2	site_no		EDD_File	Samp_No	Result_Ur	Analyte	Analysis	Sub_Matr	Result_Te	Result_Qu	Result	Reporting	Reporting	Percent
3	085N		C100611T	SLVC-0 T	3.146549	Mercury	TM_Merc	Surface W	<0.100			3.146549	0.2	
4	085N		C100611T	SLVC-0 T	3.146549	Calcium	ICPOE To	Surface W	116000		116000	3.146549	1250	
5	085N		C100611T	SLVC-0 T	3.146549	Aluminum	ICPOE To	Surface W	10300		10300	3.146549	250	
6	085N		C100611T	SLVC-0 T	3.146549	Iron	ICPOE To	Surface W	117000		117000	3.146549	1250	
7	085N		C100611T	SLVC-0 T	3.146549	Magnesiu	ICPOE To	Surface W	20900		20900	3.146549	1250	
8	085N		C100611T	SLVC-0 T	3.146549	Strontium	ICPOE To	Surface W	722		722	3.146549	50	
9	085N		C100611T	SLVC-0 T	3.146549	Silica (Si(	1.5	Surface W	46800		46800	3.146549	5000	
10	085N		C100611T	SLVC-0 T	3.146549	Silver	ICP-MS T	Surface W	<0.500			3.146549	2.5	
11	085N		C100611T	SLVC-0 T	3.146549	Thallium	ICP-MS T	Surface W	9.7		9.7	3.146549	5	
12	085N		C100611T	SLVC-0 T	3.146549	Copper	ICP-MS T	Surface W	490		490	3.146549	5	
13														

- Select "Next" once the errors have been corrected and continue the import process

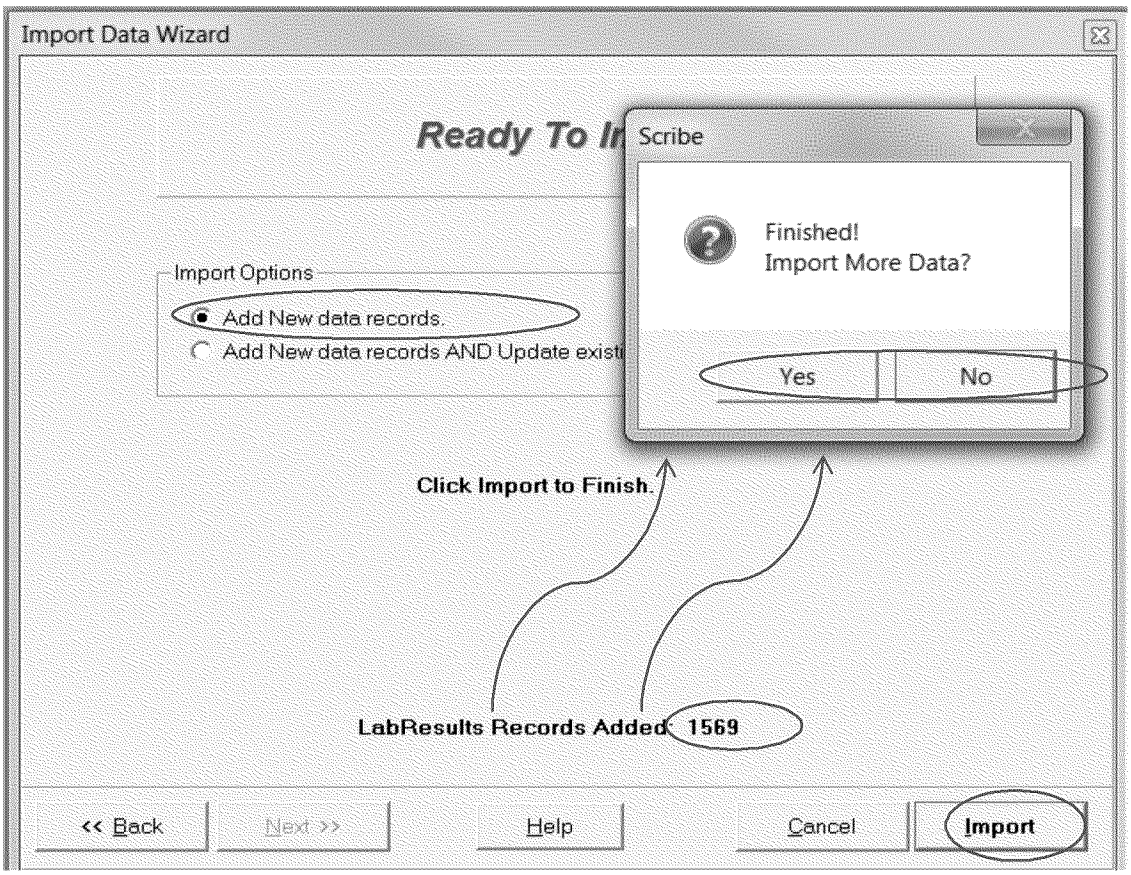
Figure 5.11 Scribe Corrected "Data to be Imported"



Samp_No	Result_Units	Analyte	Analysis	Sub_M
SLVC-0 TR	ug/L	Mercury	TM_Mercury 7470A	Surfac
SLVC-0 TR	ug/L	Calcium	ICPOE Tot. Rec Metals	Surfac
SLVC-0 TR	ug/L	Aluminum	ICPOE Tot. Rec Metals	Surfac
SLVC-0 TR	ug/L	Iron	ICPOE Tot. Rec Metals	Surfac
SLVC-0 TR	ug/L	Magnesium	ICPOE Tot. Rec Metals	Surfac
SLVC-0 TR	ug/L	Strontium	ICPOE Tot. Rec Metals	Surfac
SLVC-0 TR	ug/L	Silica (SiO2)	ICPOE Tot. Rec Metals	Surfac
SLVC-0 TR	ug/L	Silver	ICP-MS Tot. Rec. Metal	Surfac
SLVC-0 TR	ug/L	Thallium	ICP-MS Tot. Rec. Metal	Surfac
SLVC-0 TR	ug/L	Copper	ICP-MS Tot. Rec. Metal	Surfac
SLVC-0 TR	ug/L	Chromium	ICP-MS Tot. Rec. Metal	Surfac
SLVC-0 TR	ug/L	Antimony	ICP-MS Tot. Rec. Metal	Surfac
SLVC-0 TR	ug/L	Manganese	ICP-MS Tot. Rec. Metal	Surfac

- Select the "Add New Records" box
- Select "Import"
- Move the "Finished!" dialogue box away from the center portion of the "Import Data Wizard" dialogue box so that the "LabResults Records Added" total can be viewed
- Ensure that the "LabResults Records Added" is the same as the total in the "Data to be Imported" dialogue box (Figure 5.11)
- Select "Yes" if more EDDs for the same TO and Project will be loaded
- Select "No" if complete

Figure 5.12 Scribe "Import More Data?"



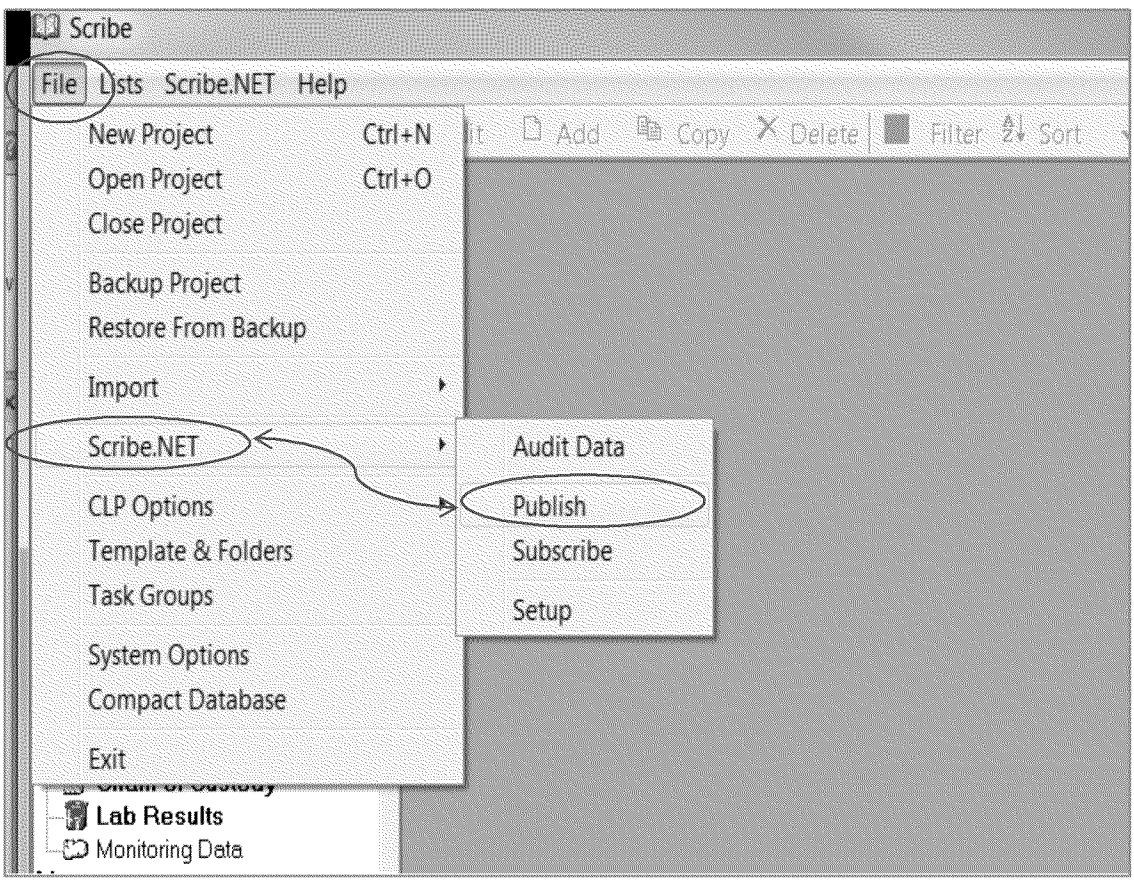
- Continue with all completed EDDs (those noted as ready to upload in the LIMS Tracking spreadsheet)

5.3 Publish Databases in Scribe

Once all EDD uploads for a specific project are loaded to Scribe, the project can be published to Scribe.NET.

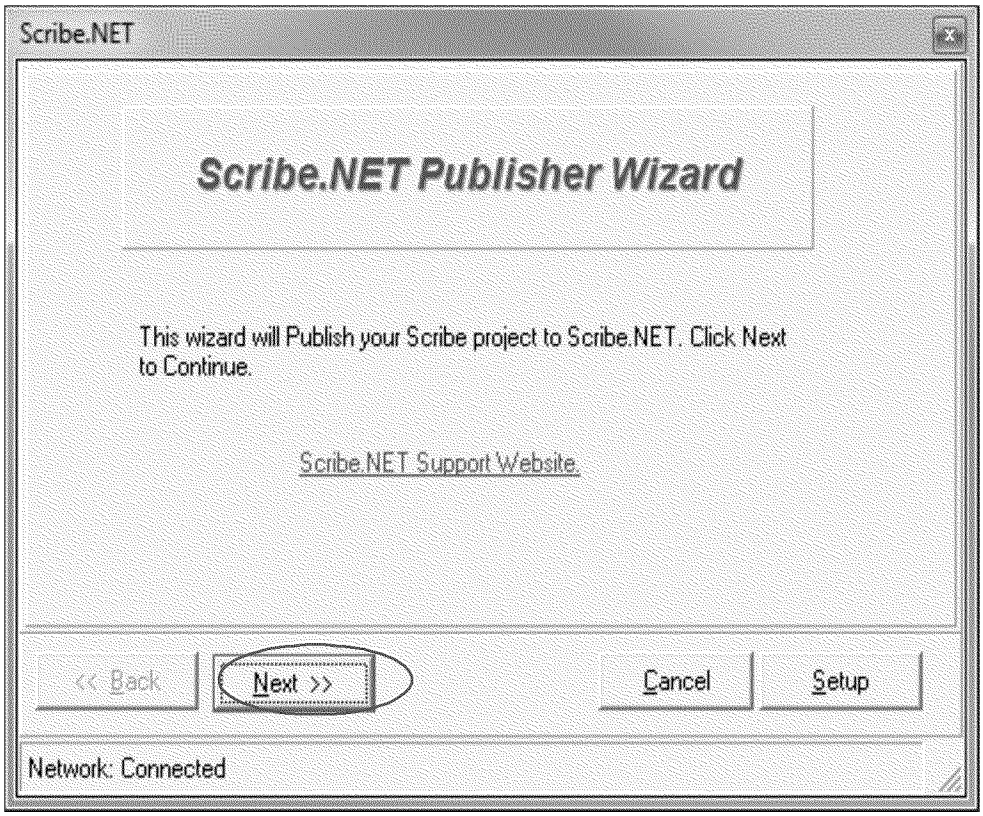
- Navigate to the LIMS Tracking spreadsheet on the network drive as described in Section 5.1 and shown in Figure 5.1
- Open the LIMS Tracking spreadsheet
- Follow the steps for opening a specific database as shown in Section 5.2, Figure 5.3, Figure 5.4, and Figure 5.5
- Select "File ->Scribe.net->Publish"

Figure 5.13 Scribe "File/Scribe.NET/Publish"



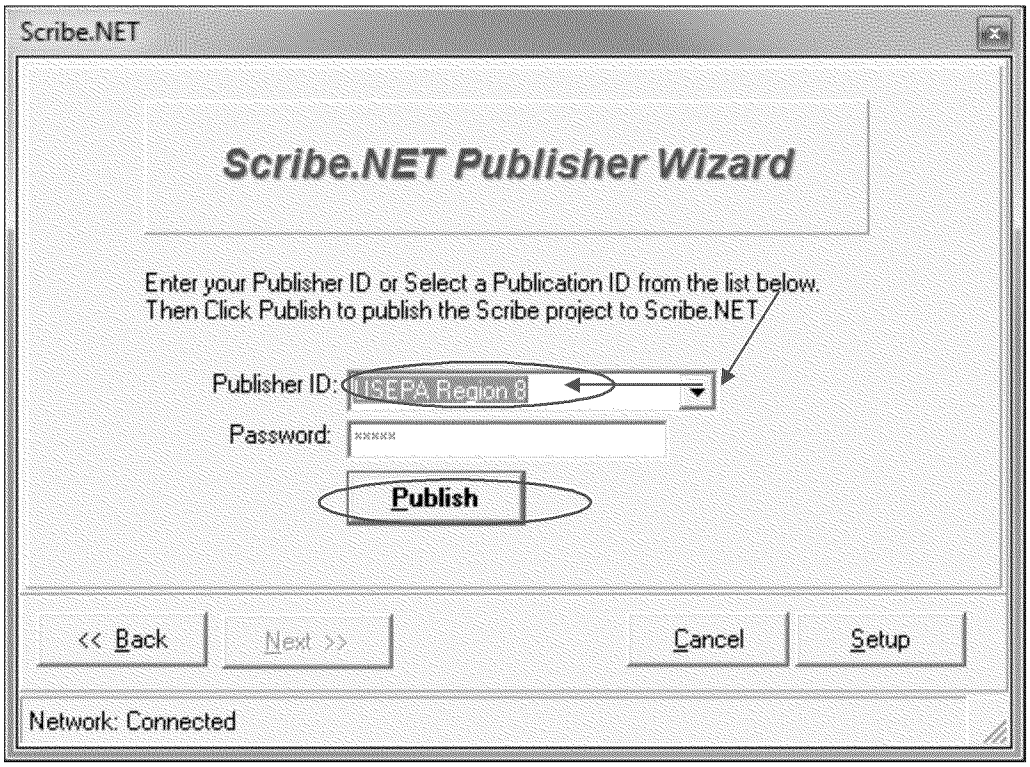
- Select "Next" on the Scribe.NET Publisher Wizard dialogue box

Figure 5.14 "Scribe.NET Publisher Wizard"



- Select "USEPA Region 8" from the dropdown list on the resulting dialogue box; leave the password field as is
- Select "Publish"

Figure 5.15 Scribe Publish Dropdown

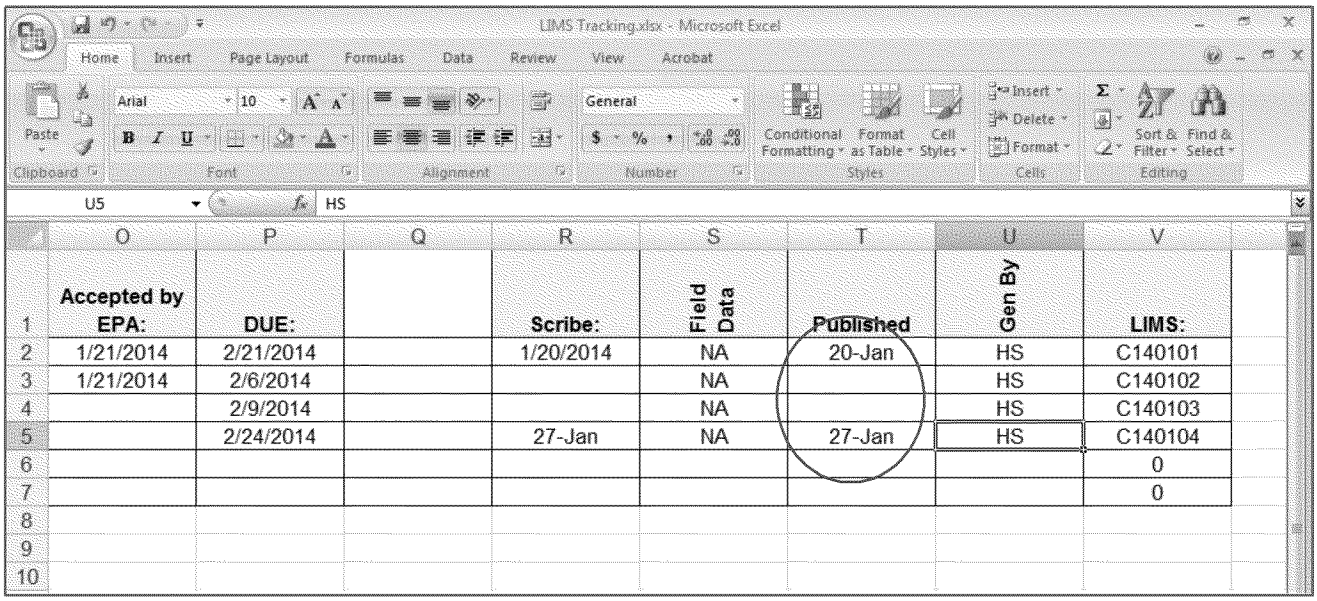


- Select "OK" in the "Finished! Project Published to Scribe.NET!" dialogue box

- **Figure 5.16 Scribe.NET "Project Published"**

- Move completed and published project folders to the correct TO subfolder titled "Final Folder" (located on the network drive, as described in Section 5.1)
- Open the LIMS Tracking spreadsheet and record the Publish date (date format defaults to spreadsheet formatting) for each Sample Event completed
- Continue with remaining TO Sample Events

Figure 5.17 LIMS Tracking Spreadsheet Input Published Date



	O	P	Q	R	S	T	U	V
1	Accepted by EPA:	DUE:		Scribe:	Field Data	Published	Gen By	LIMS:
2	1/21/2014	2/21/2014		1/20/2014	NA	20-Jan	HS	C140101
3	1/21/2014	2/6/2014			NA		HS	C140102
4		2/9/2014			NA		HS	C140103
5		2/24/2014		27-Jan	NA	27-Jan	HS	C140104
6								0
7								0
8								
9								
10								

6.0 Data Records and Management

The Data Package Coordinator will combine documents contained within each specific TO and Project folder and create and assemble the final Data Package for submittal to the client. Both the Excel and .csv versions of the EDD, as well as the data package and other associated documents, will be located in the appropriate TO Project file and Sample Event folder as shown in Figure 5.3.

7.0 Quality Control and Assurance

This SOP meets all the requirements of the ESAT Quality Management Plan.

8.0 References

ESAT Region 8, SOP,16-QAQ-03.00, Document Control, effective November 11, 2013.

ESAT Region 8, Quality Management Plan, version 7, effective June 2013.

TechLaw, Inc., Health and Safety Program Plan, effective November 2013.

TechLaw Inc., Corporate Quality Management Plan, effective November 2013.

United States Environmental Protection Agency, Environmental Response Team Software Support, accessed online at: http://www.ertsupport.org/scribe_home.htm. February 7, 2014.

United States Environmental Protection Agency Guidance for Preparing Standard Operation Procedures, EPA QA/G-6, April 2007.

Document Change History			
Revision No.	**Status[1] (I, R, C)**	**Effective Date**	**Changes Made**
0	I	03/12/14	Initial Document

[1] Status: I = Initial, R= Revision, or C = Cancelled

Soil Sampling

APPROVED:

ESAT Region 8 QA Coordinator Date

ESAT Region 8 Team Manager Date

EPA Task Order Project Officer Date

DCN: EP8-6-6111

This document has been prepared for the Environmental Protection Agency by the TechLaw, Inc. ESAT Region 8 Team and is intended to provide documentation of administrative, analytical and quality control procedures used in the daily performance of EPA and ESAT support services.

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1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to provide a guideline for the collection of representative soil samples in the field. The collection and analysis of soil samples serves to establish whether pollutants are present in the soils and helps determine the required action level(s) with regard to public and environmental health and welfare.

2.0 SCOPE AND APPLICATION

These are standard operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and included with the final report. Mention of trade names or commercial products does not constitute Techlaw, inc. endorsement or recommendation for use.

3.0 SUMMARY OF METHOD

Soil samples may be collected using a variety of methods and equipment. The methods and equipment used are dependent on the depth of the desired sample, the type of sample required (disturbed vs. undisturbed), and the soil type. Near-surface soils may be easily sampled using a spade, trowel, and scoop. Sampling at greater depths may be performed using a hand auger, continuous flight auger, a trier, a split-spoon, or a backhoe if necessary.

4.0 ACRONYMS AND DEFINITIONS

COC Chain of Custody
GPS Global Positioning System
HASP Health and Safety Plan
HAZWOPER Hazardous Waste Operations and Emergency Response
OSHA Occupation Safety and Health Administration
QA Quality Assurance
QAPP Quality Assurance Project Plan
SAP Sampling and Analysis Plan
SOP Standard Operating Procedure
EPA United States Environmental Protection Agency
ESAT Environmental Services Assistance Team

Chain of Custody (COC) – A chronological document that tracks transfer of samples between entities from collection to disposal

Composite Sampling – Sampling from several points or intervals and consolidating them into a larger sample

Discrete Sampling - Sampling from a single location

Health and Safety Plan (HASP) – A site-specific plan that outlines potential hazards and procedural/equipment recommendations

Standard Operating Procedure (SOP) - A set of written instructions that document a routine or repetitive activity followed by an organization (EPA, 2007)

5.0 HEALTH AND SAFETY

When working with potentially hazardous materials or in hazardous situations, personnel must understand and comply with the site-specific Sampling and Analysis Plan and Quality Assurance Project Plan (SAP/QAPP) and Health and Safety Plan (HASP) before the sampling event begins. More specifically, when sampling waste rock piles or fluvial deposit zones containing known or suspected hazardous substances, adequate personal protective equipment such as nitrile gloves, safety glasses, and protective footwear are necessary to prevent exposure.

When traversing tailings piles, hazardous situations exist that require the sampling personnel to wear adequate safety equipment including gloves and non-slip footwear. Never perform sampling activities if it cannot be done so in a safe manner (tailing piles are too steep, lightning is occurring, etc).

6.0 CAUTIONS

There are cautions to be considered before deployment on a soil sampling event. If the samples are to be collected in an urban area at depth, the underground utility lines must be identified. In addition, if sampling at a remote waste rock pile, always use the buddy system when traversing steep gradients that may present fall hazards. Always review the site-specific HASP for potential safety hazards.

7.0 INTERFERENCES

There are two primary interferences or potential problems associated with soil sampling. These include cross-contamination of samples and improper sample collection. Cross-contamination problems can be eliminated or minimized through the use of dedicated sampling equipment. If this is not possible or practical, then decontamination of sampling equipment is necessary. Improper sample collection can involve disturbance of the matrix (i.e. walking on specific areas that will ultimately be sampled) resulting in compaction of the sample or inadequate homogenization of the samples where required, resulting in variable, non-representative results.

8.0 PERSONNEL QUALIFICATIONS

Any personnel involved with field sampling activities must be cleared for health and safety. Clearance includes medical monitoring, respirator fit testing, and Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) 40-hour training. Personnel who will be collecting soil samples should familiarize themselves with this and other pertinent SOPs such as the Sample Equipment Decontamination SOP FLD 02.00, the Sample Preservation SOP FLD 03.00, the Sample Custody and Labeling SOP FLD 11.00, and the General Field Sampling Protocols SOP FLD 12.00.

9.0 EQUIPMENT

Equipment needed for collection of soil samples may include:

HASP Gear - Gloves, proper footwear, safety glasses, etc.

Mapping and Location Tools – Global Positioning System (GPS) units, site/local area maps, compass, tape measure, survey stakes, pin flags, camera, 2-way radios

Documentation Tools – Field log book, field data sheet, COC(s), labels, clear tape, pens, permanent marker, waterproof paper

Sampling Tools – Plastic, Teflon™, or other appropriate composition scoop (analysis dependent), shovel, spade, trowel, measuring cup or graduated cylinder, field scale, bucket auger, post hole auger, homogenization container w/ mixing tool, bucket, rinse bottle, purified water, paper towels

Sample Containers – Ziploc™ baggies, glass jars (organics analysis), labels, clear tape, pens, permanent marker, cooler(s), ice, thermometer

See Appendix A for a detailed list of soil sampling equipment.

10.0 STANDARDS AND REAGENTS

Reagents are not used for the preservation of soil samples. Decontamination solutions are specified in the Sampling Equipment Decontamination SOP# FLD-02 and the site-specific work plan.

11.0 PROCEDURES

11.1 Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies required.

2. Obtain the necessary sampling and monitoring equipment.

3. Decontaminate or pre-clean equipment, and ensure that it is in working order.

4. Prepare schedules, and coordinate with staff, client, and regulatory agencies where necessary. It is also important to obtain access agreements if sampling is to occur on private property.

5. Perform a general site survey prior to site entry in accordance with the site-specific HASP.

6. Use stakes, flagging, or buoys to identify and mark all sampling locations followed by a GPS point (see GPS Trimble® GeoXT 2008 series SOP FLD 07.00). Specific site factors, including extent and nature of contaminant should be considered when selecting sample location. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. All staked locations will be utility-cleared by the property owner

prior to soil sampling. It is the responsibility of the sampler to verify with the property owner that utility lines have been marked. If there is no property owner and there is concern for underground utility lines, it is the responsibility of the samplers to contact the state agency or contractor that can provide a marking service.

11.2 Sample Collection

In general, there are two primary ways to collect a soil sample. Composite sampling involves taking several subsamples from a designated sample location and consolidating into one larger sample. Discrete sampling is defined as taking one sample from a single location. Composite and discrete sampling can be achieved by the sample techniques listed below.

11.2.1 Surface Soil Samples

Collection of samples from near-surface soil can be accomplished with tools such as spades, shovels, trowels, and scoops. Remove surface material to the required depth and use a stainless steel or plastic scoop to collect the sample.

This method can be used in most soil types but is limited to sampling near-surface areas. Accurate, representative samples can be collected with this procedure depending on the care and precision demonstrated by the sample team member. A stainless steel scoop, lab spoon, or plastic spoon will suffice in most other applications. The use of a flat, pointed mason trowel to cut a block of the desired soil can be helpful when undisturbed profiles are required. Care should be exercised to avoid use of devices plated with chrome or other materials. Plating is particularly common with garden implements such as potting trowels. There are four depth classes that are typically used in Region 8: 0-2” range, 0-6” range, 6-12” range, and 12-18” range. The 0-2” and 0-6” range can usually be sampled with one of the tools listed above, but the deeper ranges generally require the use of one the tools described in sections 11.2.2 and 11.2.3.

The following procedure is used to collect surface soil samples:

1. Carefully remove the top layer of soil or debris to the desired sample depth with a pre-cleaned spade.

2. Using a pre-cleaned, stainless steel scoop, plastic spoon, or trowel, remove and discard a thin layer of soil from the area which came in contact with the spade.

3. If volatile organic analysis is to be performed, transfer the sample directly into an appropriate, labeled sample container with a stainless steel lab spoon or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval or location into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the

caps tightly.

4. Due to data quality requirements, some soil sampling events may require that each sub-sample of a composite be measured. This can be achieved two ways: by mass or by volume. Due to the remote nature of the sites in Region 8, it is recommended that composite samples are measured by volume. This requires the use of a measuring cup or graduated cylinder (of appropriate composition), placing the material into the measuring device to the desired volume, then adding the sub-samples to a larger sample container (plastic baggie for metals, glass jar for organics). Overall volume of sample will be dictated by analytical requirements.

11.2.2 Sampling at Depth with Augers and Thin Wall Tube Samplers

This system consists of an auger, or a thin-wall tube sampler, a series of extensions, and a " T" handle (Figure 1, Appendix B). The auger is used to bore a hole to a desired sampling depth and is then withdrawn from the hole. The sample may be collected directly from the auger. If a core sample is to be collected, the auger tip is then replaced with a thin wall tube sampler. The sampling assembly is then lowered down the borehole, and driven into the soil to the completion depth. The system is withdrawn and the core is collected from the thin wall tube sampler.

Several types of augers are available; these include: bucket type, continuous flight (screw), and post-hole augers. Bucket type augers are better suited for direct sample recovery since they provide a large volume of sample in a short time. When continuous flight augers are used, the sample can be collected directly from the auger flights. The continuous flight augers are satisfactory for use when a composite of the complete soil column is desired. Post-hole augers have limited utility for sample collection as they are designed to cut through fibrous, rooted, swampy soil and cannot be used below a depth of three feet.

The following procedure is used for collecting soil samples with the auger:

1. Attach the auger bit to a drill rod extension, and attach the "T" handle to the drill rod.

2. Clear the area to be sampled of any surface debris (e.g., twigs, rocks, litter). It may be advisable to remove the first three to six inches of surface soil for an area approximately six inches in radius around the drilling location.

3. Begin augering, periodically removing and depositing accumulated soils onto a plastic sheet spread near the hole. This prevents accidental brushing of loose material back down the borehole when removing the auger or adding drill rods. It also facilitates refilling the hole, and avoids possible contamination of the surrounding area.

4. After reaching the desired depth, slowly and carefully remove the auger from the boring. When sampling directly from the auger, collect the sample after the auger is removed from the boring and proceed to Step 10.

5. Remove the auger tip from drill rods and replace with a pre-cleaned thin wall tube

sampler. Install the proper cutting tip.

6. Carefully lower the tube sampler down the borehole. Gradually force the tube sampler into soil. Care should be taken to avoid scraping the borehole sides. Avoid hammering the drill rods to facilitate coring as the vibrations may cause the boring walls to collapse.

7. Remove the tube sampler, and unscrew the drill rods.

8. Remove the cutting tip and the core from the device.

9. Discard the top of the core (approximately 1 inch), as this possibly represents borehole debris material collected before penetration of the layer of concern. Place the remaining core into the appropriate labeled sample container. Sample homogenization is not required.

10. If volatile organic analysis is to be performed, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

11. If another sample is to be collected in the same hole, but at a greater depth, reattach the auger bit to the drill and assembly, and follow steps 3 through 11, making sure to decontaminate the auger and tube sampler between samples.

12. Abandon the hole according to applicable State regulations. Generally, shallow holes can simply be backfilled with the removed soil material.

11.2.3 Sampling at Depth with a Trier

The system consists of a trier, and a "T" handle. The auger is driven into the soil to be sampled and used to extract a core sample from the appropriate depth. The following procedure will be used to collect soil samples with a sampling trier:

1. Insert the trier (Figure 2, Appendix B) into the material to be sampled at a 0 to 45 angle from horizontal. This orientation minimizes the spillage of sample.

2. Rotate the trier once or twice to cut a core of material.

3. Slowly withdraw the trier, making sure that the slot is facing upward.

4. If volatile organic analysis is to be performed, transfer the sample into an appropriate,

labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

11.2.4 Sampling at Depth with a Split Spoon (Barrel) Sampler

The procedure for split spoon sampling describes the collection and extraction of undisturbed soil cores of 18 or 24 inches in length. A series of consecutive cores may be extracted with a split spoon sampler to give a complete soil column profile, or an auger may be used to drill down to the desired depth for sampling. The split spoon is then driven to its sampling depth through the bottom of the augured hole and the core extracted.

When split spoon sampling is performed to gain geologic information, all work should be performed in accordance with ASTM D 1586-67 (reapproved 1974). The following procedures will be used for collecting soil samples with a split spoon:

1. Assemble the sampler by aligning both sides of barrel and then screwing the drive shoe on the bottom and the head piece on top.

2. Place the sampler in a perpendicular position on the sample material.

3. Using a well ring, drive the tube. Do not drive past the bottom of the head piece (past the full length of the sample barrel) or compression of the sample will result.

4. Record in the site logbook or on field data sheets the length of the tube used to penetrate the material being sampled, and the number of blows required to obtain this depth.

5. Withdraw the sampler, and open by unscrewing the bit and head and splitting the barrel. The amount of recovery and soil type should be recorded on the boring log. If a split sample is desired, a cleaned, stainless steel knife should be used to divide the tube contents in half, longitudinally. This sampler is typically available in 2 and 3 1/2 inch diameters. However, in order to obtain the required sample volume, use of a larger barrel may be required.

6. Without disturbing the core, transfer it to appropriate labeled sample container(s) and seal tightly.

11.3 Sample Sieving

Analytical methods may require that a sample be separated by particle size. A sieve is the most effective method of separating coarse and fine material from a soil sample. Sieving and random

sampling is also an effective method for soil sample homogenization (Schumacher et al, 1990). The site-specific SAP should be consulted when deciding what particle size of a soil sample should be submitted for analysis. Note that saturated soil samples should not be sieved. Those samples must first be dried before processing. Sieving procedures:

- Place sample in appropriate sized sieve (dry samples are optimal; wet samples will stick to the grid of a sieve).
- Use a catch pan that is of a material that won't compromise the integrity of the sample.
- Place lid on the sieve and shake vigorously to separate particle sizes.
- Transfer desired sample fraction to labeled container.
- Decontaminate thoroughly with brushes and/or compressed air before use on next sample.

12.0 DATA RECORDS AND MANAGEMENT

Once collected, samples are preserved, labeled, and stored for transport. A COC must accompany all samples during transport and transfer between entities. Sample labels should contain the following information:

- Site Identification
- Date sampled
- Sampler initials
- Time
- Analysis to be performed

In addition, soil characteristics may need to be documented when sampling. Below is a standardized list of soil characteristics and their corresponding Unified Soil Classification System identifiers.

- GW – well-graded gravels, gravel and sand mixtures, little or no fines
- GP – poorly graded gravels, gravel and sand mixtures, little or no fines
- GM – silty gravels, gravel, sand, silt mixtures
- GC – clayey gravels, gravel, sand, clay mixtures
- SW – well-graded sands, little or no fines
- SP – poorly-graded sands, little or no fines
- SM – silty sands, sand-silt mixtures
- SC – clayey sands, sand-clay mixtures
- ML – inorganic silts and very fine sands
- CL – inorganic clays of low to medium plasticity
- OL – organic silts and organic silty clays
- MH – inorganic silts, micaceous or diatomaceous fine sandy or silty soils
- CH – inorganic clays of high plasticity
- OH - organic clays of medium to high plasticity, organic silts
- Pt – highly organic soils

13.0 QUALITY CONTROL AND ASSURANCE

There are no specific quality assurance (QA) activities which apply to the implementation of these procedures. However, the following QA procedures apply:

1. All data must be documented on field data sheets or within site logbooks.

2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. An instruction manual for the operation of the hand auger equipment is provided in Appendix C of this SOP. Equipment checkout and calibration activities must occur prior to sampling/operation, and they must be documented.

3. Always consult the SAP for duplicate sample frequency requirements.

4. Document any deviations from SOP's, work plan, SAP/QAPP, etc.

14.0 REFERENCES

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Appendix A

Soil Sampling Equipment

Table 9.0-1 Soil Sampling Equipment

Category	Item	Use	Comment
Health and Safety	Gloves	Protection from absorption of contaminants	Nitrile are recommended
Health and Safety	Boots	Slip/contaminant protection, warmth	Any type are acceptable
Health and Safety	Safety Glasses	Eye protection	Sunglasses for UV protection
Health and Safety	Layered Clothing	Protection from hypothermia	polyester base layers only
Mapping/Location	GPS unit	Sample station locating	Pre-loaded with site locations
Mapping/Location	Maps	Location identification	must contain most current information
Mapping/Location	Two-way radios	Communication	extra batteries or charger required
Documentation	Field logbook	Site data and conditions documentation	waterproof pages
Documentation	Chain of Custody	Sample handling/identification	Pre-printed using Scribe
Documentation	Labels	Sample Identification	Pre-printed using Scribe
Documentation	Clear tape & scissors	Label protection	
Sampling Tools	Bucket/Transfer device	Sample transfer (if required)	Can also be used for sample homogenization
Sampling Tools	Scoop, spade or trowel	Surface soil sampling	select based on analysis
Sampling Tools	Auger	Depth soil sampling	consult SAP sample objectives
Sampling Tools	Cooler	Sample containment	no ice for soil samples
Sampling Containers	Amber glass jars	Volatile Organics Analysis sample containment	tight cap seal
Sampling Containers	Ziploc™ baggies	Metals analysis sample containment	double bag if necessary
Reagents	10% Nitric Acid (HNO3) solution	For decontamination of metals sampling equipment	Pre-mixed at lab
Reagents	10% Hydrochloric (HCl) acid solution	For decontamination of organics sampling equipment	Pre-mixed at lab

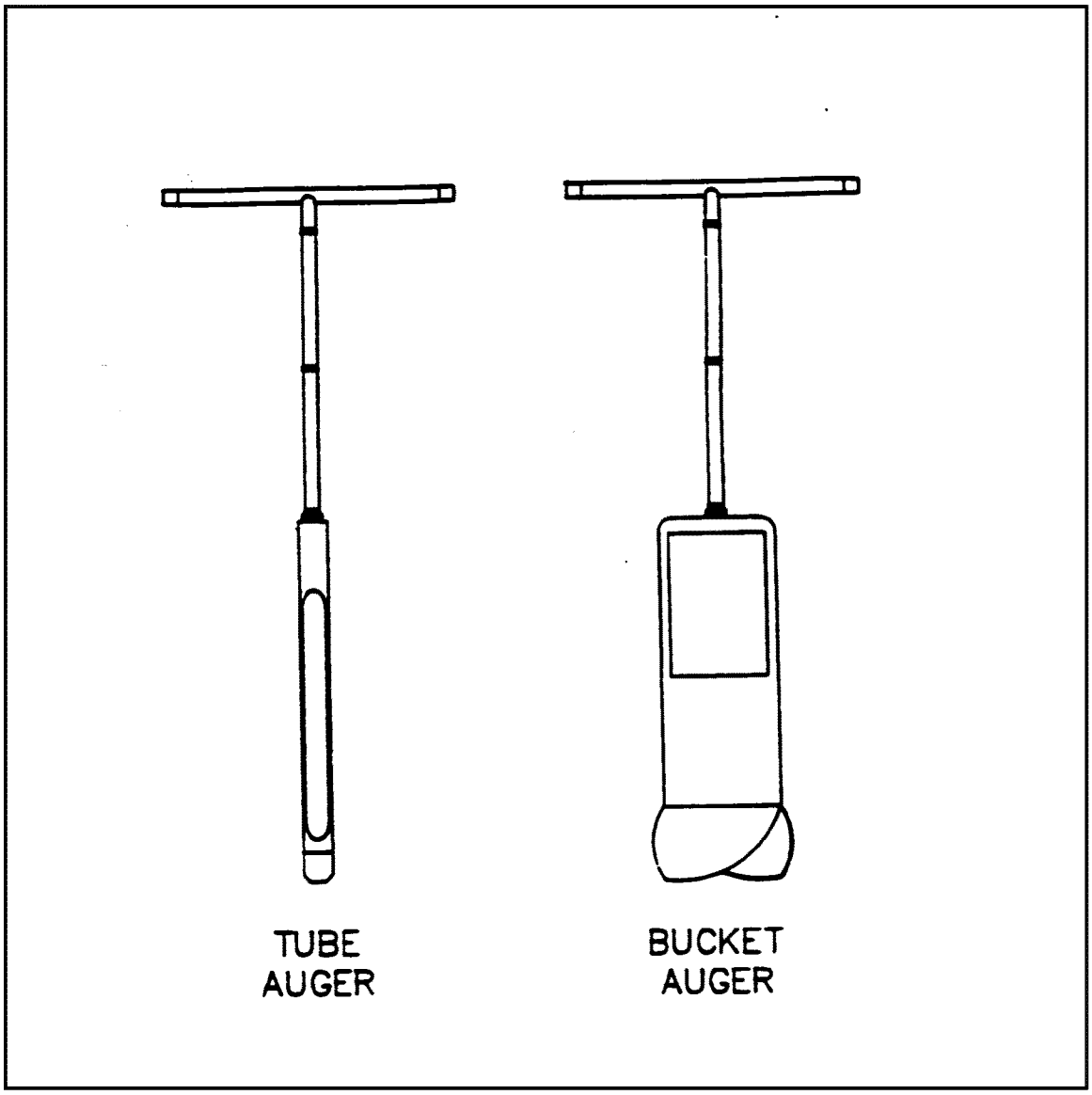
Appendix B

Auger and Trier Diagrams

APPENDIX A

Figures

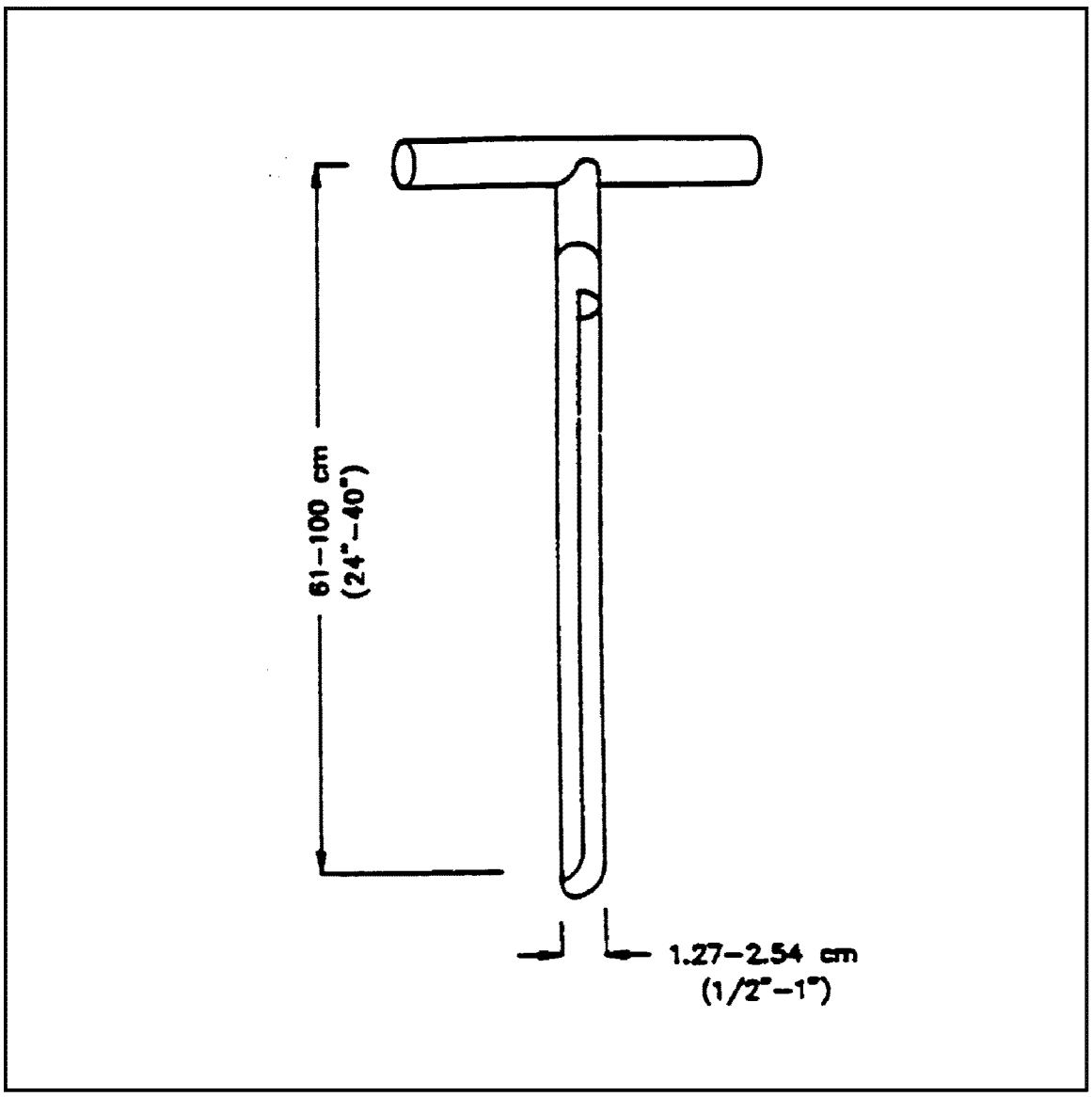
FIGURE 1. Sampling Augers



APPENDIX A(Cont'd)

Figures

FIGURE 2. Sampling Trier



Appendix C

Hand Auger Operating Instructions

OPERATING INSTRUCTIONS

01.11.SO HAND AUGER SET FOR HETEROGENEOUS SOILS

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P.O. Box 4, 6987 ZG Giesbeek, NL
T +31 313 880200
F +31 313 880299
E eijkelkamp@eijkelkamp.com

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On these operating instructions

If the text follows a mark (as shown on the left), this means that an important instruction follows.

If the text follows a mark (as shown on the left), this means that an important warning follows relating to danger to the user or damage to the apparatus.

Introduction

The hand auger set for heterogeneous soils consists of a number of types of augers held in a transport case. An auger consists of a bottom part with auger body to which a handle and optional extension rods can be attached. Contrary to the single type auger this set is suitable for almost any type of soil. It is lightweight and easy to manipulate by a single person.

Upon erosion of rock soils are formed and transported by natural processes to be deposited elsewhere. Soils consist of minerals, organic material and cavities filled with air and/or water. The minerals may vary in size of clay and loam (< 63 μm) and sand (63 μm – 2 mm), to gravel (2 - 63 mm) and stones (> 63 mm).
Soil in its natural position may consist of various types of soils (stratified or heterogeneous soil). Differences in texture or soil-forming processes lead to stratification. Soils type will vary from clay, loam, sand to stony soil, depending on its loam and sand content. Peaty soil mostly consists of organic material.
Cohesive soils are wet clay, loam and peaty soil. Sand and stony soils are moderately cohesive.

The hand auger set for heterogeneous soils is suitable for manual augering and sampling in a wide variety of soil types for the purpose of general soil research (profiles, geology and mineralogy) and sampling in environmental research. Depending on the water table, structure and type of soil, augering can be done up to a depth of 5 m. Augers are available for any type of soil above or below the water table, and may be selected to adjust to the situation at hand. It is possible to switch augers in the process (see 5. The use of augers).

1. Description

The hand auger set for heterogeneous soils is available as a full standard set for augering up to 5 m of depth, containing bottom parts and various auger bodies, extension rods, handles, a push/pull handle, utility probe, maintenance kit and various accessories in an aluminium transport case (see picture, page 1). The full set weighs 26.4 kg.

1.1 Edelman auger

The Edelman auger body (see picture, next page) is conical in shape and consists of two blades (1) joined in a bit (2). The top of the blades is welded to a bracket (3), which is connected to the auger rod (4). The blades are vaulted and when entering the soil the sample is dug up and evenly guided into the inside of the auger body. The vaulting of the blades not only promotes digging up but also ensures a firm grip of the sample while permitting easy emptying of the auger body.

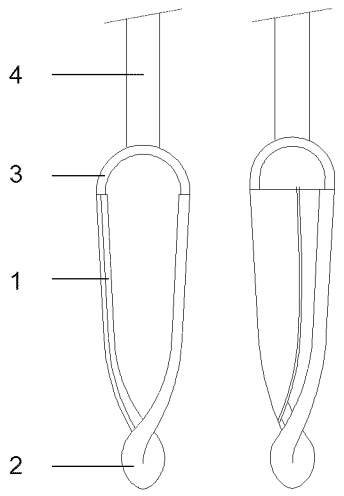
All Edelman augers in the set have an identical diameter (measured diagonally between the blades at the broadest part of the auger body). The various Edelman augers vary in blade breadth and shape of bit, viz.:

Clay type
Combination type
Sand type
Coarse sand type

The Edelman auger, clay type.
Clay soils can be very cohesive; therefore, the blades of the clay type can be narrow. The blades meet with little resistance permitting augering to be done with a minimum of effort, producing a hardly disturbed sample. Wider blades would cause extra friction and the soil material would be difficult to remove.
The Edelman auger, combination type.
The blades of this type are slightly broader and rounder than those of the clay type, permitting a good hold of moderately cohesive soils, while cohesive soils can easily be removed. The bit is elongated allowing easy augering in stiffer soil.

The Edelman auger, sand type.
This type of Edelman auger is suitable for moderately cohesive soils, such as sandy soils. The elongated, twisted bit ensures easy entering. The blades of the sand type are broader than those of the combination type allowing a good hold of the soil.

The Edelman auger, coarse sand type.
This auger is based on the sand type but the blades have extra wings thus forming an almost closed auger that will firmly keep hold of loose soils such as coarse sand or very dry sandy soils.



Edelman auger combination type (left) and coarse sand type (right)

1.2 Riverside auger

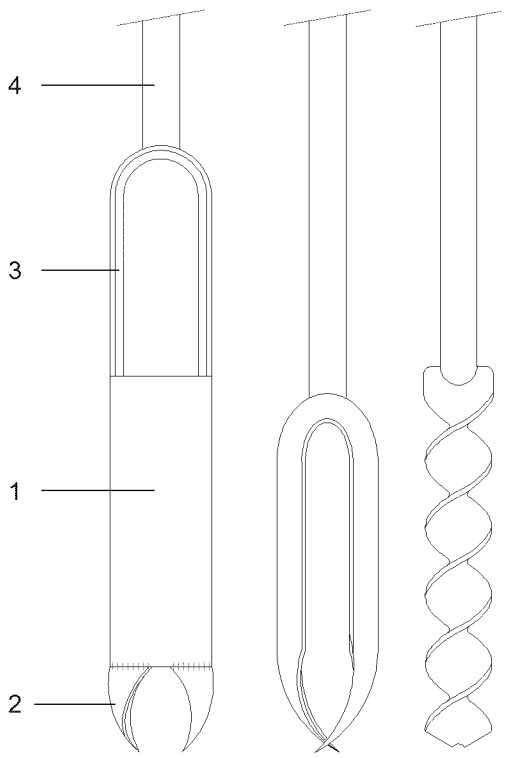
Dry and hard soils do not permit the use of the Edelman auger with its centric bit. Therefore, the Riverside auger is being used (see figure). The auger body has an open tube (1) with two beak-like bits at its base (2) and a bow bracket (3) at its top, which connects to the auger rod (4). The extremities of the bits are skewed and scrape the soil, pressing the sample evenly into the tube. The diameter of the auger bits slightly exceeds the tube's diameter so as to reduce friction between soil and tube to a minimum.

1.3 Auger for stony soils

The auger for stony soils is heavier and more rigid than the other types of augers. It has pointed cutting bits, which are bent inward but not touching, rendering certain flexibility. The bits cut downward thus gripping and holding the stones and other less coarse soil material (such as gravel holding coarse sands) between the blades.

1.4 Spiral Auger

Where other augers cannot cope with hard and rigid soils, the spiral auger is ideal (see figure). Its narrow spiral with its negative end (the centre is whetted away, and the two ends are bent) easily digs into the soil pushing stones aside. Its straight design causes friction when hoisting the auger.



Riverside auger (left), stony soil auger (middle) and spiral auger (right)

1.5 Piston Sampler

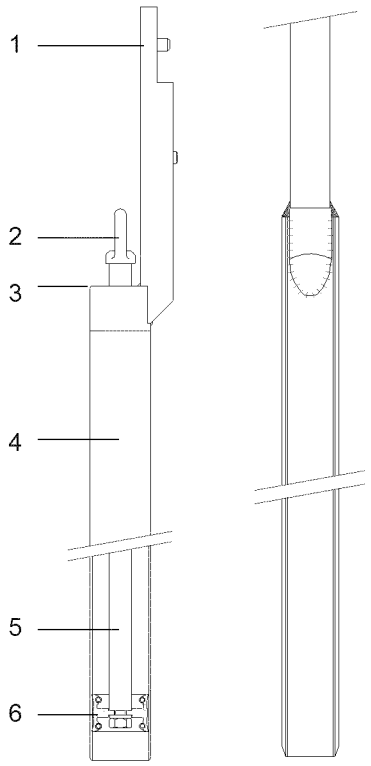
The piston sampler (see figure) is constructed from a thin-walled, 4 cm diameter, stainless steel tube (4). The bottom end is open, whereas the top has a lit (3) through which a stainless steel piston rod (5) can be moved, and outflow openings. The bottom end of the piston rod holds a piston (6), and to its top-end a wire eye (2) is attached. The top-end of the tube is equipped with a bayonet connection (1) welded on its outside to permit free passage of the piston rod. Due to the eccentricity of the sampler one has to reckon with a maximum of 6 cm when working with auger holes. The maximum length of the sample is 75 cm.

1.6 Gouge Auger

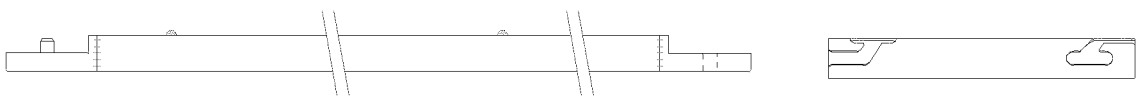
The body of a gouge auger is half-cylindrical, with parallel cutting edges running from top to bottom. The auger body is attached to the bottom part at its top end. Its diameter is smaller than that of other augers. The maximum sample size is 50 cm.

1.7 Accessories

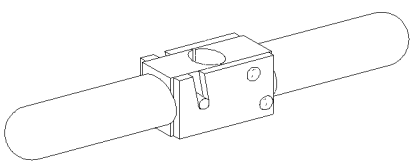
Upper part, extension rods and coupling sleeves.
The upper part measures 60 cm and has a detachable, synthetic grip. The extension rods measure 1 m. Since the upper part, the extension rods and bottom parts have bayonet connections, the auger can be adjusted to any length. Coupling sleeves (see figure) are cylindrical and lock the connections.



Piston sampler (left) and gouge auger (right)


Extension rod (left) and coupling sleeve (right)

The Push/Pull Handle.
The push/pull handle has two parts that can be fitted around a rod. Once pressure is exercised on the two bars of the handle its construction ensures a firm hold on the rod.

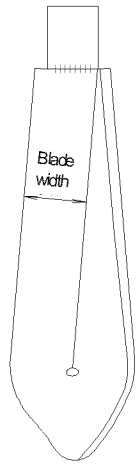


Utility Probe.
The fibreglass utility probe measures 105 cm, and has a 19 mm cone diameter. It is insulated allowing safe checking of the substratum for cables, tubes and pipes.

2. Technical specifications

Various important dimensions of different types of augers are listed in the table on the next page. Diameters have been established by diagonally measuring the widest point between the blades.

Type of auger	Diameter (mm)	Blade width (mm)
Edelman clay type auger	70	30
Edelman combination type auger	70	35
Edelman sand type auger	70	40
Edelman coarse-sand type auger	70	75
Riverside auger	70	N/A.
Stony soil auger	70	N/A.
Spiral auger	40	N/A.
Piston sampler	Tube 40	
	Total 6 cm	N/A.
Gouge Auger	30	N/A.



The auger bodies are manufactured in (non-toxic, not stainless steel) iron-manganese steel. The piston sampler is made of stainless steel. All auger bodies are unpainted for the purpose of environmental research.

3. Safety instructions

Hold the coupling sleeve in the middle, this will prevent you from catching the skin of your hands between the parts while (dis)connecting them.

Prior to augering use the utility probe to check for cables, tubes and pipes. If necessary, select another spot to auger.

While augering hold the auger by its synthetic handle. It is fully insulated should you hit an electricity cable.

Augers over 4 m should be handled in parts. This will prevent damage to the rods and reduce the risk of being hit by augers tipping over. This applies to inserting and hoisting the auger.

Do not force, or pound on, the auger. This may cause serious damage, such as cracks or snapped joints.

After augering fill up the borehole with soil or special bentonite plugs. This will prevent humans or animals to trip into the hole and incur injuries, and restores impermeable soil layers.

Be cautious during a thunderstorm. Lightning strokes often occur in the open field, in particular when one holds metal objects.

4. Preparing for use

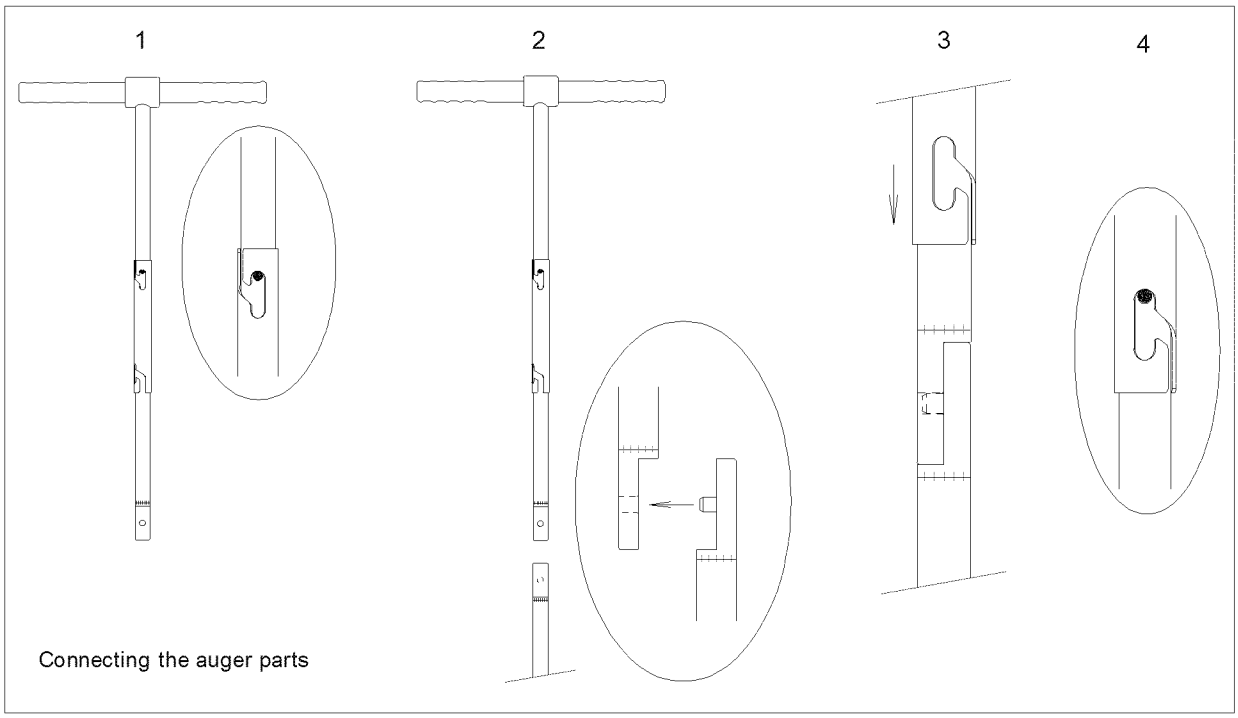
1. Detach the coupling sleeves from the extension rods and the upper part.

2. Screw the handle into the upper part.

3. Select the appropriate auger (see 6. Application). The Edelman combination type auger often proves to be the best choice.

4. Connect the auger parts (see figure on the next page).

   4.1 Hold the coupling sleeve in the middle and slide it onto the upper part until it clicks on the nipple (step 1). The sleeve is locked when it cannot be rotated.



Connecting the auger parts

4.2 Join the upper and bottom part (step 2).

4.3 To lock the connection, unscrew the sleeve from the upper part, and slide it across the connection (step 3) and click it onto the nipple (step 4). Check the lock. Notice it will have a slight play.

Hold the coupling sleeve in the middle, this will prevent you from catching the skin of your hands between the parts while (dis)connecting them.

5. The use of augers

5.1 General instructions

Each type of auger has its specific application. However, the instructions below apply to all augers.

Prior to augering use the utility probe to check for cables, tubes and pipes. If necessary, select another spot to auger.

While augering hold the auger by its synthetic handle. It is fully insulated should you hit an electricity cable.

Rotate the auger clockwise.

Hold the auger perfectly vertical while drilling.
Over 1,20 m extension rods should be used:

1. Place the auger flat on the ground near the borehole.
2. Slide the sleeve off the bottom part, and lock it onto the upper part.
3. Detach the upper and bottom part.
4. Select an extension rod and a sleeve. Lock the sleeve over the bayonet at the bottom end of the extension rod (the open end).
5. Attach the upper and bottom part to the extension rod.

Always check the coupling sleeves. Well-attached sleeves will prevent jamming or loss of parts when augering.

An auger over 4 m should be inserted and hoisted in parts:

1. Insert the auger in the borehole and place the bottom part on end for approximately 50 cm. Grip the auger firmly!
2. To attach: connect the two parts, and slide the sleeve of the upper part across the connection and lock it onto the bottom part.
   To detach: slide the sleeve upward and lock it onto the upper part. Unclamp the upper and bottom part.

Augers over 4 m should be handled in parts. This will prevent damage to the rods and reduce the risk of being hit by augers tipping over. This applies to inserting and hoisting the auger.

The auger's depth is indicated by marks. Small nipples on the bottom part indicate 50 cm from the bottom end of the auger. The piston sampler has no marking and the gouge auger has a small nipple at 75 cm.
The upper part has a larger nipple indicating a depth of 1 m, except for the piston sampler and gouge auger, however, where it indicates a depth of 1.35 and 1.25 m respectively. Additional meters depend on the number of extension rods.
To obtain an undisturbed sample rotate the auger clockwise without pushing, this will cause the sample to detach from the soil.
To withdraw the auger after sampling, pull it upward while turning it (not when using the Riverside auger or the auger for stony soils). Keep your back straight and your knees bent to prevent injuries. Wear gloves for a full grip on the rods.
Place the samples in a logical order of augering to allow examination.
Should you encounter various types of soils, switch to other types of augers (see 6. Application).

Do not force, or pound on, the auger. This may cause serious damage, such as cracks or snapped joints.

After augering fill up the borehole with soil or special bentonite plugs. This will prevent humans or animals to trip into the hole and incur injuries, and restores impermeable soil layers.

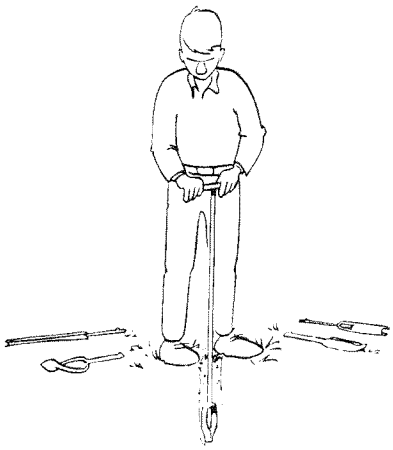
Be cautious during a thunderstorm. Lightning strokes often occur in the open field, in particular when one holds metal objects.

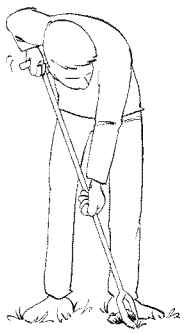
Clean the parts of the auger by rinsing them after use. This will prevent jamming of the sleeves or the piston sampler to leak (see 8. Maintenance).

5.2 Edelman auger

Hold the auger by its handle and rest it on the ground (see figure, next page). Rotate it clockwise while gently pushing it into the soil. Upon 2¼ complete rotations of 360° the auger should have dug 10 cm. The auger body will be filled up to its bracket with slightly disturbed soil material. Depending on the type of soil additional rotations may be necessary.

Withdraw the auger after sampling; hoist it while gently rotating the auger. To release the cohesive material hold the auger askew on the surface (see figure, next page), rotate the auger 180° while pressing it into the ground. The sample should detach itself and can be taken out by hand or by lightly tapping the auger. Moderately cohesive material will detach itself immediately. Now, examination is possible.





Augering (left), emptying (middle) and examination (right)

Caution:

Do not overfill the auger body. Superfluous material will coat the auger hole, which hinders pulling out subsequent soil samples. When augering under the water table an overfilled auger acts like a plunger, which hampers hoisting the auger and results in loss of sample material.
Loss of sample material. Hoist the auger with sample while lightly rotating it, do not pull it straight out.

5.3 Riverside auger and auger for stony soils

Screw the auger into the soil. The soil will be fed evenly into the body. Pull out the auger without rotating it. Tap it gently on the surface and the sample will detach itself. In cohesive soils it is difficult to empty the Riverside auger, consequently another type (the Edelman auger) is recommended.

5.4 Spiral auger

Push and turn the auger into the soil. The auger body will push the stones aside. Hoist the auger lightly rotating it. In certain types of soils its design may cause considerable friction. The sample will often be disturbed. The spiral auger is often used to penetrate hard layers before other type of augers can be used.

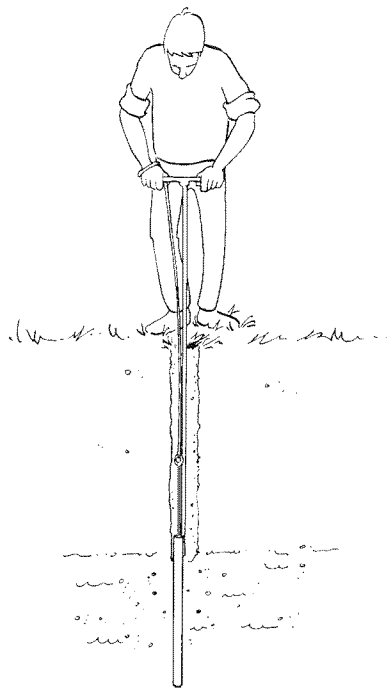
5.5 Piston sampler

1. Attach a polyester cord to the piston sampler's wire eye and let the (extended) auger down to the bottom of the augered hole. The piston rod should remain in the lowest position. By shaking the piston rod it will fall to its lowest position.

2. Pull the cord attached to the wire eye of the piston rod and an underpressure will be created below the piston.

3. Push the tube steadily down (see figure) while keeping the cord (and the piston) stationary, i.e. at a constant distance to the sample material.

Pressing down the tube may cause resistance. Small pulling movements of the cord will cause an increase of the underpressure, thus reducing resistance and disturbance of the sample.



4. When the tube has been filled, push once more and pull it out of the auger hole.

To keep the sample in the tube, the piston should remain in the highest position by keeping the cord taut (if necessary, tie the cord to the handle). Keep the piston rod parallel with the auger rod to prevent the piston to leak, and consequently loss of sample.

5. Place the sampler horizontally on the surface and push the sample out of the tube with the piston. Shaking the tube will facilitate this process. The sample will have a 75 cm undisturbed profile.

Remarks:

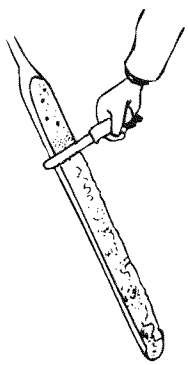
In the case of cohesive soils it is necessary to auger a hole up to a moderately cohesive layer under the water table using another type of auger. Thin and cohesive strata (such as clay or loam up to a maximum of several centimetres) within a moderately cohesive layer may cause clogging of the tube This impedes pressing the tube into the soil. It is recommended to note the depth of the cohesive layer. Pushing out the sample may cause it to flow as a result of increased water pressure behind the cohesive layer, thus disturbing the sample.

The piston sampler should be used to sample one auger hole at a time. After sampling the auger hole may cave in and cannot be augered further.

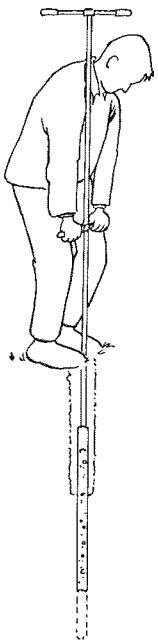
If the difference in height between the water level in the auger hole and the surface is too large, the sample may flow out of the tube. Filling the auger hole with water can prevent this.

5.6 Gouge auger

Push the gouge auger vertically into the soil without rotating it. Take a sample of maximally 50 cm. Should you encounter resistance completely rotate the auger and press again.



Cut off the sample by completely rotating the auger without pressing down and hoist it gently. Use a bent spatula to cut off protruding soil along the cutting edges (see figure), this will provide an almost undisturbed profile. If necessary the sample can be marked every 10 cm using the marks on the outside of the gouge auger. Wear sturdy gloves emptying the gouge auger. Push out the sample using the bent spatula, the convex part upwards.



When augering at greater depth, attach extension rods to the upper end of the gouge auger, directly below the upper part. The rods tend to bend following the line of the auger hole. Follow the same order when (dis)connecting the rods.

5.7 Accessories

Push/pull handle.

The push/pull handle is ideal for insertion or withdrawal of the auger without straining your back (see figure). It is clamped around the extension rods at any desired height.

Two persons may also use the push/pull handle. To that purpose clamp the handle perpendicularly to the direction of the auger's top handle. Face the other person holding the bars of the push/pull handle with your right hand and holding the bars of the top handle with your left hand exerting up-or downward pressure.

Sounding device.

The sounding device is used to determine the ground water level in the auger hole. Drop it into the auger hole until it hits the water surface with a 'plopping' sound. The depth is crucial in determining the choice of auger. Note that, depending on the type of soil, it may take some time for the water in the auger hole to level with the water table.

6. Application

The auger set for heterogeneous soils is suitable for augering in almost any type of soil (not in solid rock or very stony soils). There are various auger types for any type of soil. In heterogeneous soils one can switch type of auger. The augers in the set can be used for drilling up to a depth of 5 m, depending on depth of the ground water, soil structure and type of soil.

The Edelman auger can be used in various types of soil. In homogeneous soil one of the four types will suffice. In unidentified or mixed soil types the combination auger will be preferred. In hard or stony soils the Riverside auger or auger for stony soils or spiral auger will be useful. The piston sampler is suitable in sandy soils under the water table, giving an undisturbed profile. The gouge auger also provides an undisturbed profile and is suitable for soft, cohesive soils, above and below the water table.

The table below lists the application of various auger types.

Types of auger	Application
Hand auger set	Soil research in almost any type of soil.
Edelman clay type	Marshy or clay soils above the water table.
Edelman combination type	Universal: clay soils below the water table.
Edelman sand type	Sandy soils above the water table.
Edelman coarse sand type	Coarse sand soils above the water table.
Riverside auger	Hard, rigid soils, such as dry clay soils; fine stony soils.
Stony soil auger	Stony soils, gravely soils containing small stones.
Spiral auger	Very hard, rigid soils such as iron pans, chalk and lime profiles. Particularly suitable to drill through, or to pre-auger in combination with other types of augers.
Piston sampler	Sandy soils below the water table or in open water.
Gouge auger	Soft, cohesive soils such as clay, loam and peaty soil.

7. Troubleshooting

Soil particles between the augering rod and the coupling sleeve have caused the sleeve to jam. Pour clean water in one direction in the sleeve, this will flush out the particles. Use the synthetic backside of the spatula to tap the sleeve, coarse particles will become come loose thereby allowing the sleeve to slide. In winter conditions icing up may cause the sleeve to jam.

Augering is strenuous. This may be caused by a faulty match of auger and type of soil (see 6. Application), or incorrect augering (see 5. The use of augers).

Loss of sample during augering. This may be caused by a faulty match of auger and type of soil (see 6. Application), or incorrect augering (see 5. The use of augers).

The piston sampler leaks, leading to loss of sample. Slide the piston rod up and down parallel with the auger rod to prevent the piston to dislocate. Non-parallel movements can damage the piston permanently. Grains of sands may cause damage and leakage to the piston. Damaged pistons should be replaced (see 8. Maintenance).

Loss of sample material. The difference in height between the water level in the auger hole and the surface is too large. Fill the auger hole with water.

Make sure not to lose the coupling sleeves. Count them after augering. Carry them attached to an extension rod or to the upper part. Always check whether the sleeves are locked. Two spare sleeves are provided.

Make sure to withdraw every single part of the auger. Always check whether the sleeves are locked.

The sounding device does not produce a 'plopping' sound when passing the water table. Move it quickly up and down to increase its downward speed. Make a rough estimation of the depth of the water table in the tube and increase accuracy upon every examination.

8. Maintenance

It is recommended to keep the augering equipment in good condition by rinsing it during use. Flush out any dirt from the piston sampler by moving the piston rod up and down under water.

Clean the augers after use with running water. Take off the coupling sleeves from the rods and the upper parts, clean and dry them well to keep the insides smooth and prevent oxidation (rough inner surfaces of the sleeve may cause it to jam). To avoid excessive oxidation when storing the auger body, apply Vaseline (not for the piston sampler).

The piston of the piston sampler can be removed for cleaning. Hold the wire eye and turn the nut on the piston using a (ring) spanner 13. Push the piston rod to remove the piston (slant it slightly) from the tube. Clean the piston, position it in the tube, insert the piston rod, place the spring washer and tighten the nut.

The auger bodies need no whetting, use keeps them sharp-edged.Under normal conditions oxidation is not detrimental to the auger and will vanish upon use.

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Global Positioning System (GPS) - Trimble GeoXT 2008 Series

APPROVED:

ESAT Region 8 QA Coordinator Date

ESAT Region 8 Team Manager Date

EPA Task Order Project Officer Date

ESAT Region 8 Task Lead Date

DCN: EP8-6-6513

This document has been prepared for the Environmental Protection Agency by the TechLaw, Inc. ESAT Region 8 Team and is intended to provide documentation of administrative, analytical and quality control procedures used in the daily performance of EPA and ESAT support services.

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standard approach for United States Environmental Protection Agency (EPA) and Environmental Services Assistance Team (ESAT) Region 8 personnel to use a Global Positioning System (GPS) during field activities.

2.0 SCOPE AND APPLICATION

This SOP is specifically intended for application by EPA and ESAT personnel who conduct field work using a GPS.

3.0 SUMMARY OF METHOD

This SOP covers the use of TerraSync on Trimble GeoXT 2008 Series GPS machines as well as the Laser RangeFinder and Pathfinder Office. This SOP is based on manufacturers' instructions.

4.0 ACRONYMS AND DEFINITIONS

EPA United States Environmental Protection Agency
ESAT Environmental Services Assistance Team
GIS Geographic Information System
GPS Global Positioning System
HASP Health and Safety Plan
SOP Standard Operating Procedure

Geographic Information System (GIS) : A system of hardware and software used for storage, retrieval, mapping, and analysis of geographic data.

Global Positioning System (GPS) : A navigational system involving satellites and computers that can determine the latitude and longitude of a receiver on Earth by computing the time difference for signals from different satellites to reach the receiver.

Terrasync: Software used in GPS units that captures and stores spatial data collected in the field.

Trimble: A private GPS Company that provides equipment and technical support, including the 2008 GeoXT GPS units and TerraSync software.

5.0 HEALTH AND SAFETY

There are no health and safety issues requiring mention in this SOP; however, refer the applicable site-specific Health and Safety Plan (HASP) any time field work is conducted.

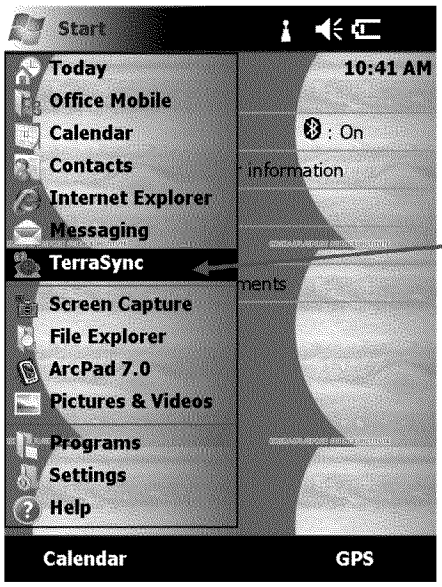
6.0 EQUIPMENT

Trimble GeoXT 2008 Series GPS unit with TerraSync
LTI TruPulse 360 Laser RangeFinder
TruPulse 200B/36B Cheat Notes card
Tape measure

Flags
Computer and/or external hard drive

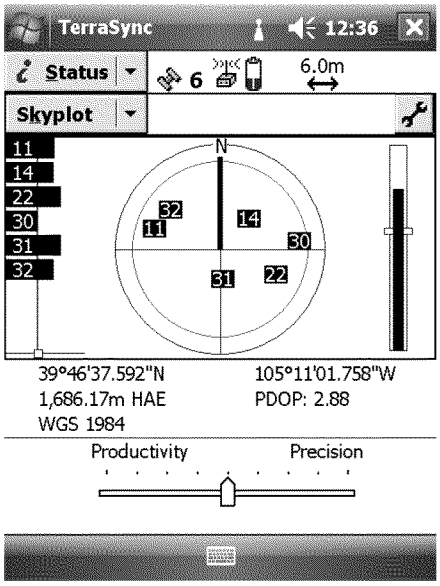
7.0 TERRASYNC

7.1 Starting TerraSync



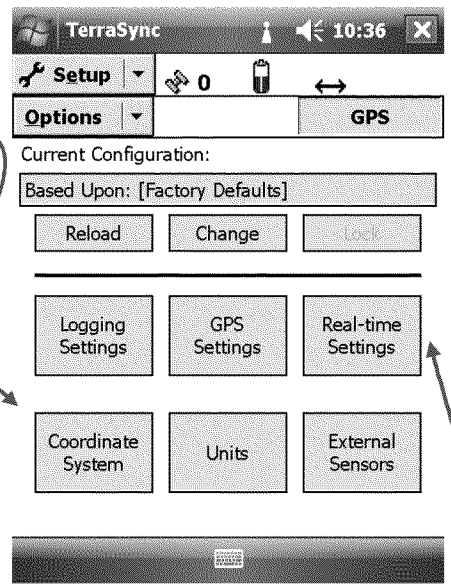
To begin, press the green button on the Trimble unit. Tap **Start**, then **TerraSync** to open the GPS application (left). If not available, tap **Programs**, then select **TerraSync**.

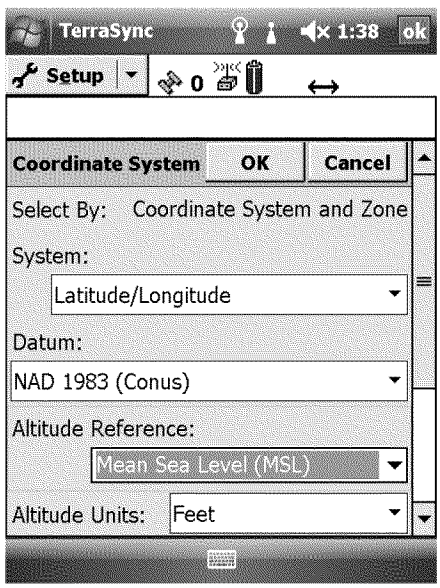
TerraSync will open with the **Status-Skyplot** page and begin to acquire satellite information. A minimum of four satellite locks are necessary to obtain a position. More satellites and proper satellite geometry increase accuracy.



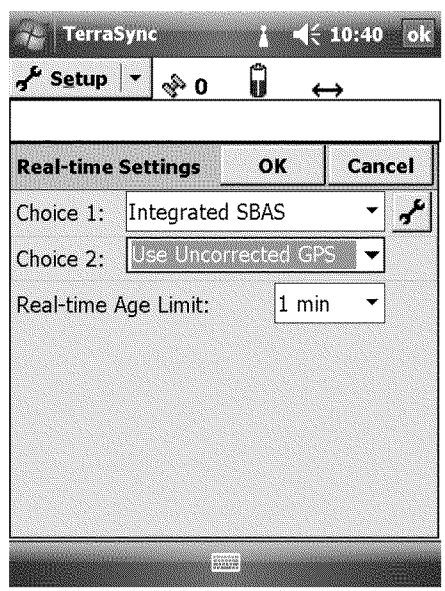
7.2 TerraSync Setup Menu

As TerraSync begins to acquire satellites, make sure the unit is properly configured. Tap the upper drop down menu and choose **Setup**. From the **Setup** page, tap **Coordinate System** and ensure that your screen matches the figure below. If a different coordinate system is required, use the dialog shown below to make necessary changes. Keep defaults if unsure. Select **OK** to return to setup menu.

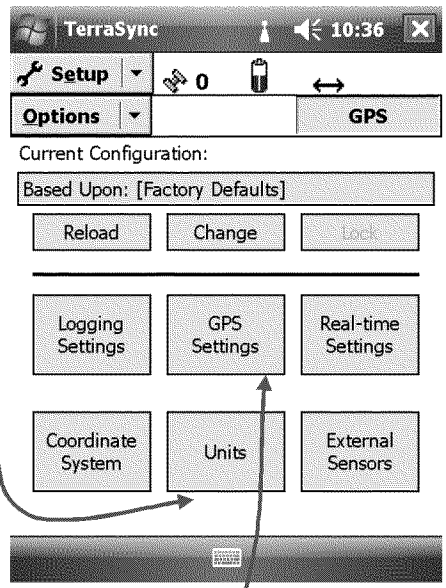


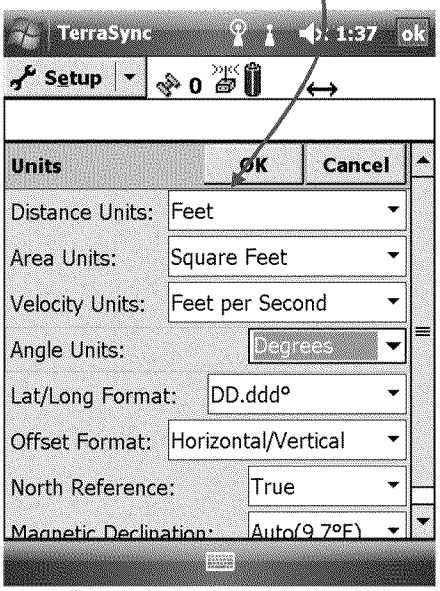


Next, choose **Real-Time Settings.** These settings will allow you to receive real time differentially corrected readings from satellite-based radio transmitters, if available. If not available, you will be using uncorrected GPS signals that can be corrected later by ESAT Region 8 GIS personnel. Please ensure that your screen matches the figure on the right. Select **OK** to return to setup menu.

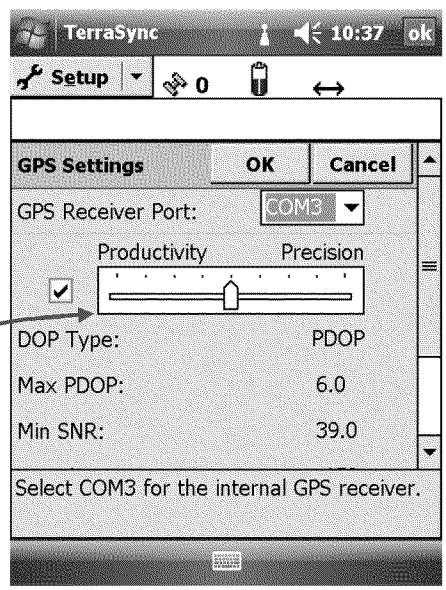


The **Units** menu allows you to choose which units will be used for display in the **Map** section (feet). Make any necessary changes. Select **OK** to return to setup menu.

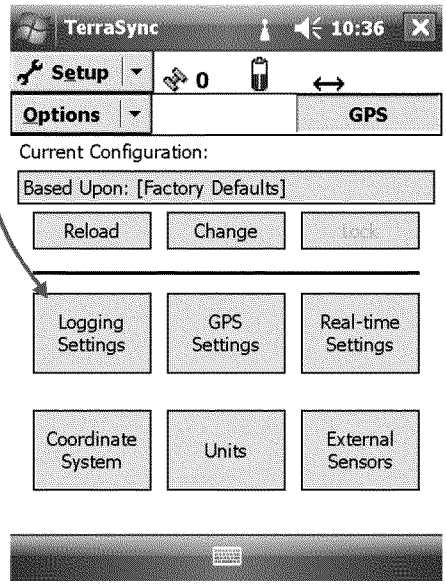


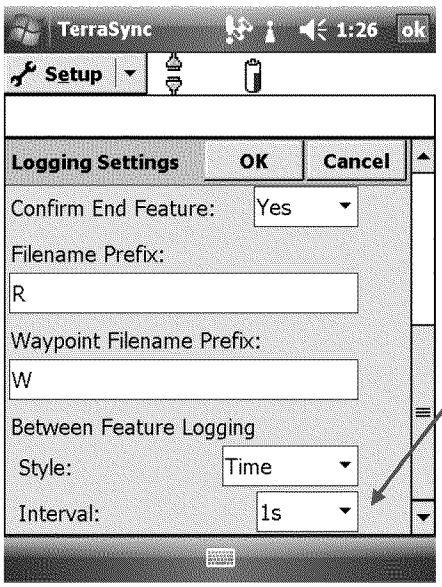


Under the **GPS Settings** menu, you can control the level of accuracy that your unit measures by adjusting the slider bar. Keep the defaults unless you are sure that you are willing to sacrifice precision for productivity or vice versa. Make sure that the **Productivity/Precision Bar** is set in the middle to give you a PDOP of 6.0, a minimum SNR of 39, and a minimum elevation of 15 degrees. Keep the settings for Velocity Filter and NMEA Output set to "Off." Select **OK** to return to setup menu.

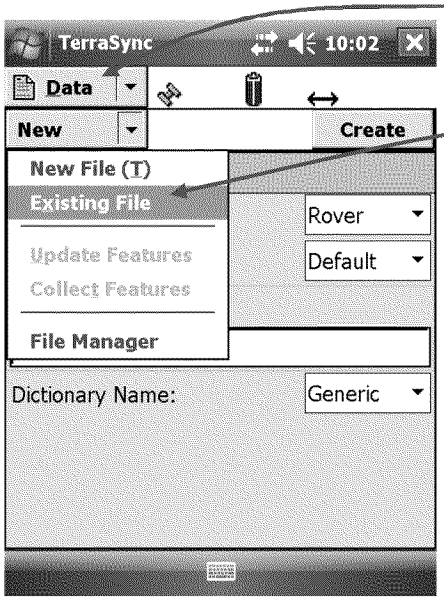


The **Logging Settings** menu allows you to adjust how you will log your data. In this dialog box, you will set your logging interval to either 1 or 5 seconds, depending on the data being collected (the default used by ESAT is 1 second; however, 5 seconds is better suited for high volume data collection, such as a large polygon or area). You will also set your antenna height to the height at which you will be collecting your data. Normally, this will be set at 1 meter (approximately 3.5 feet or the height an average person would hold the unit). If you wish to change Antenna Height, click the following icon located just right of the Antenna Height figure:

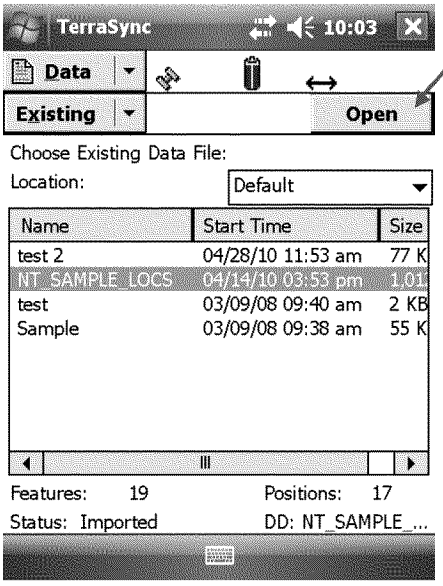




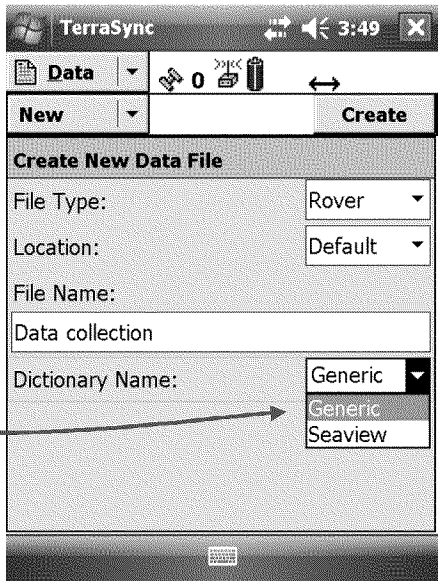
7.3 TerraSync Data Menu



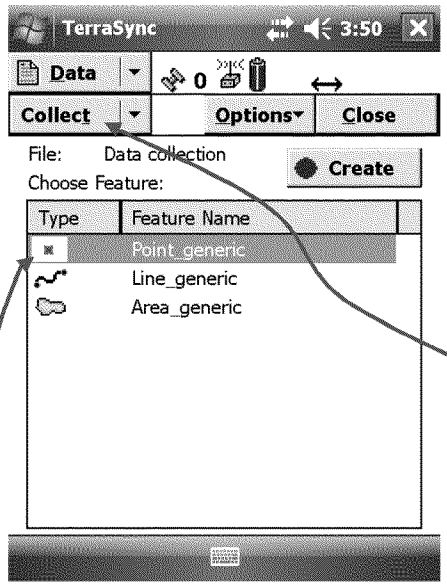
Select **Data** from the main drop down menu. The default will be to create a new data file. Click the drop down labeled **New** and choose **Existing File**. From this menu, select the appropriate file and click on the **Open** button. Then confirm antenna height.



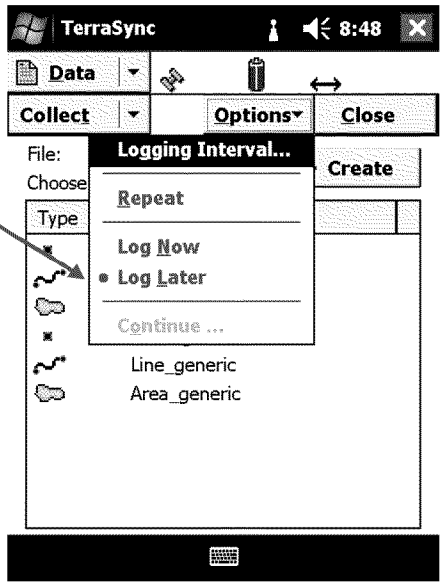
If you wish to create a new file or cannot find the appropriate existing file, click the **New** tab. Rename the new file to a project specific name. Select the proper data dictionary. Choose **Generic** if a data dictionary does not exist for your project. Tap **Create** and confirm antenna height at 1 meter (~3.5 feet) in subsequent pop-up.



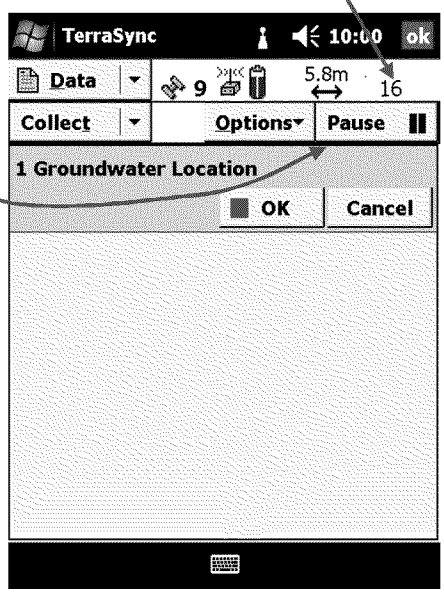
The features you are able to collect are now available in a list (below left). Click the **Options** tab and select **Log Later**. Use the **Options** tab to select a logging interval appropriate for the type of data you are collecting (below right).



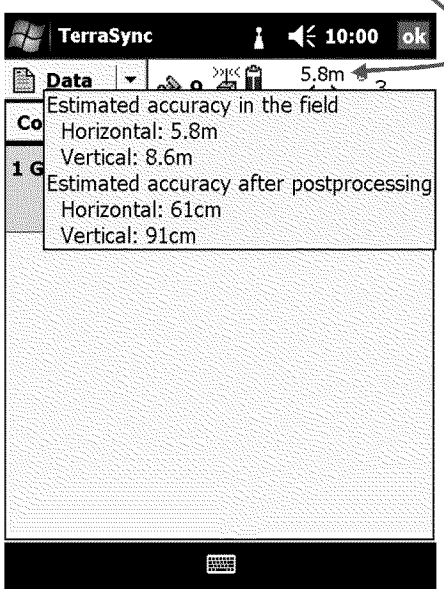
Ensure that this Tab has **Collect** selected.



Select **Point**, **Line**, or **Area,** then tap the **Create** button. When ready to collect data, tap the **Log** button (button will show pause if not currently logging, as shown here). Attribute any and all necessary fields (tap keyboard icon to enter description). This can be done before, during, or after collecting your data. Make sure to collect at least 30 positions for each point. Your positions count is displayed above the **Log** button.

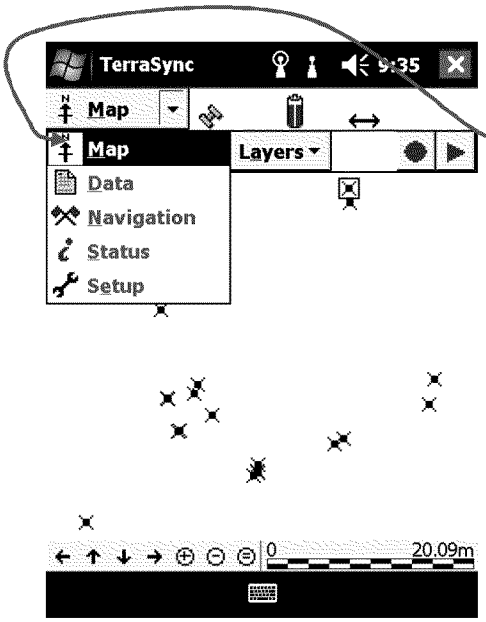


Estimated accuracy is listed here. By tapping this number more detail will be shown (right).

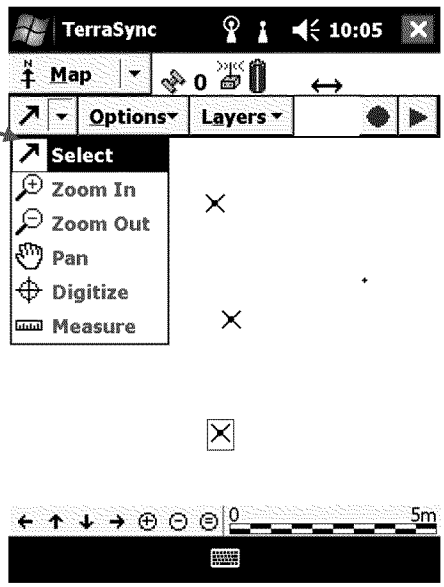


When ready, click **OK** to finish feature collection. You will be returned to the main data collection page. Continue collecting data until finished. Data will be saved as you go. When finished with data collection, exit out of TerraSync and turn off unit (Green button).

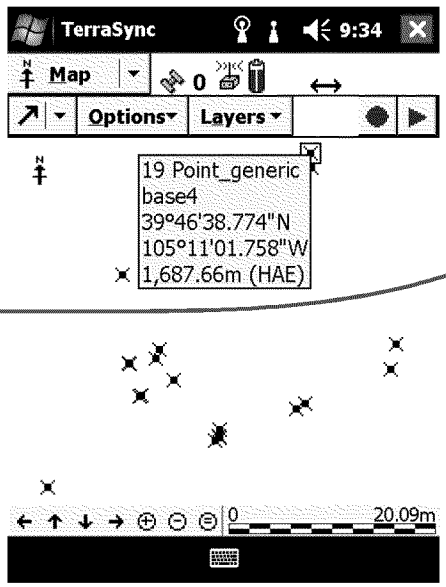
7.4 TerraSync Map Menu

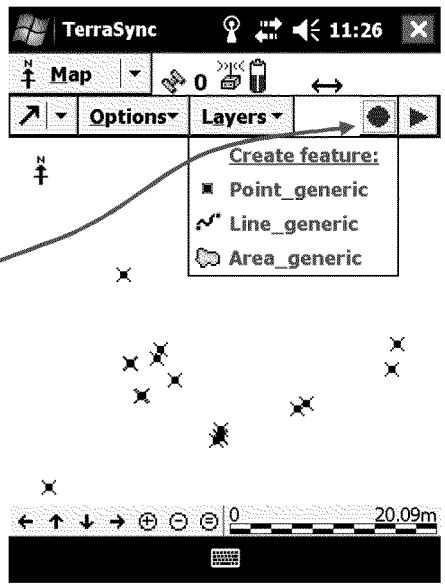


To view data that has been collected, choose the **Map** section. There are multiple tools to help with the display of data shown here.

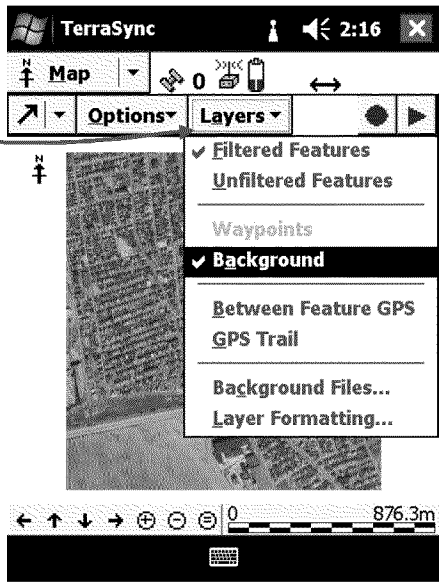


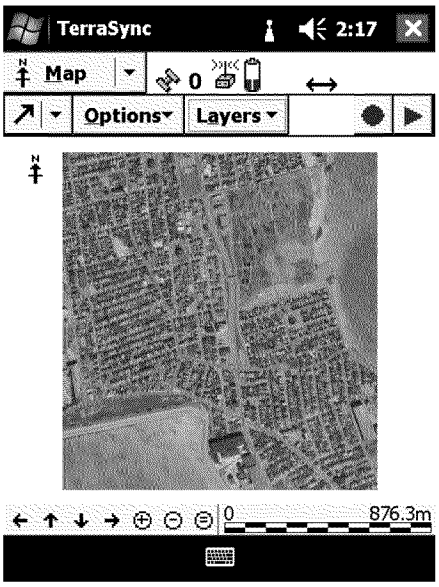
Tapping any point will display the attributes of a point, including a point's latitude and longitude. Logging GPS positions can be accomplished in the **Map** section. After selecting and setting up the proper data file in the **Data** section, choose **Map.** Tap **Create Feature** button, then tap the arrow key on the top right to begin logging points. When enough points have been collected, click **Ok**.





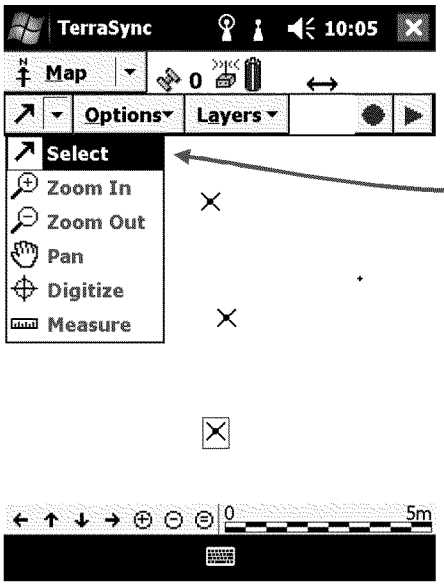
The on-screen display in the **Map** section can be enhanced with an aerial or topographical image. This must be set up prior to field collection with ESAT. If available select **Layers** and tap **Background** to enable the image (See Below).

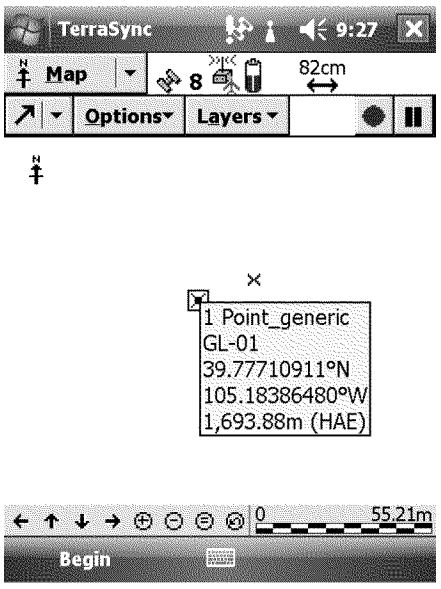




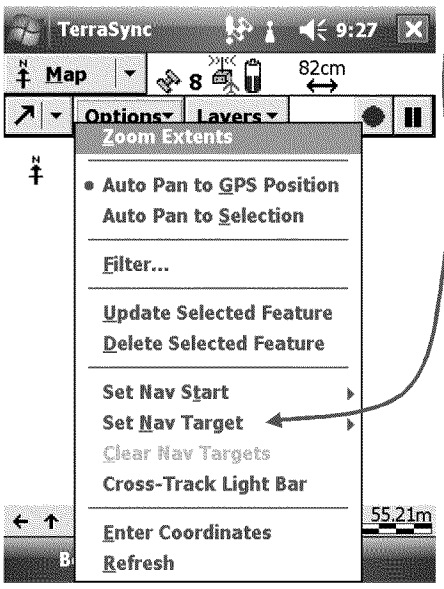
7.5 Navigation Using TerraSync

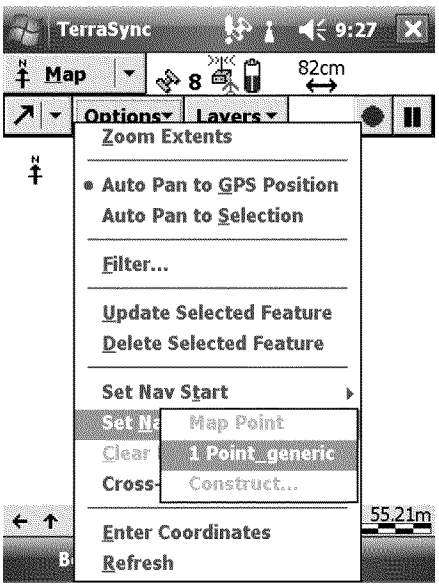
TerraSync and your Trimble unit can help you to navigate to a specific point if that point has already been loaded into the data. In the **Map** tab, use the selection arrow to select the point of interest (below, right).



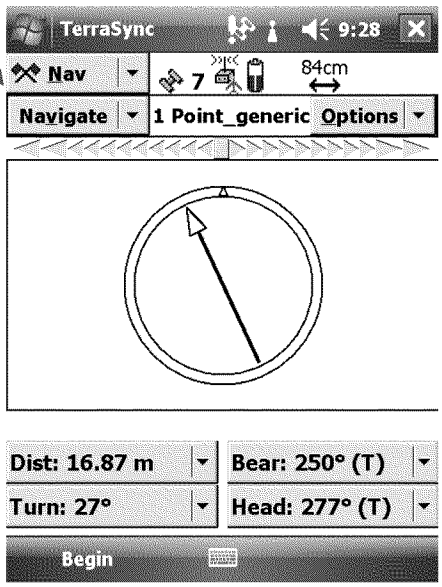


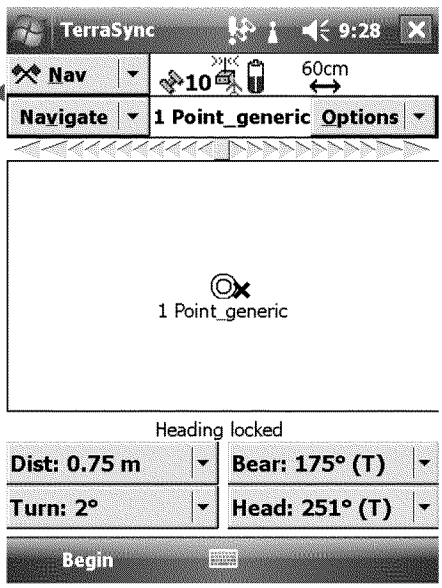
Tap the **Options** tab and then select **Set Nav Target.** Next, select the highlighted feature in the drop down menu (below). On the map, the point you are navigating to will have a crossed flag symbol like this:



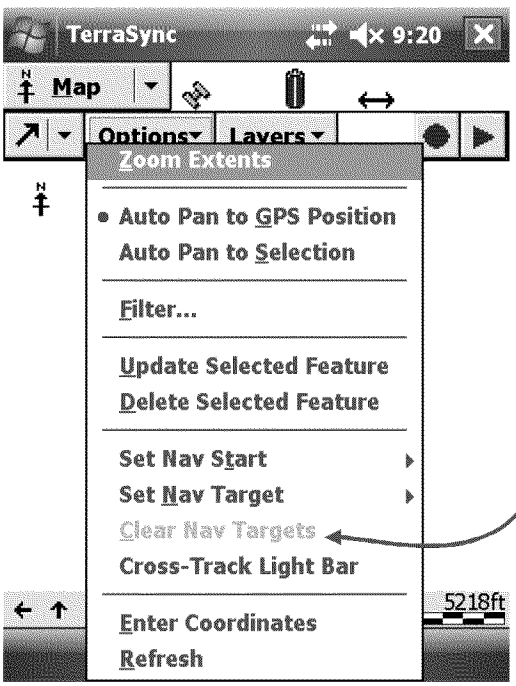


Once you have selected the point feature of interest, you can switch to the **Nav** menu on the main drop down list (left). The arrow guides you in the direction of the point of interest giving distance and bearing. You have to be moving for this to work. This navigation menu will change once you are close (right). You are the **X.**





Once finished navigating, go back to the **Map** section by going to the **Nav** menu and clicking **Map**. In the **Map** section, click **Options** and select **Clear Nav Targets.**



8.0 LASER RANGEFINDER

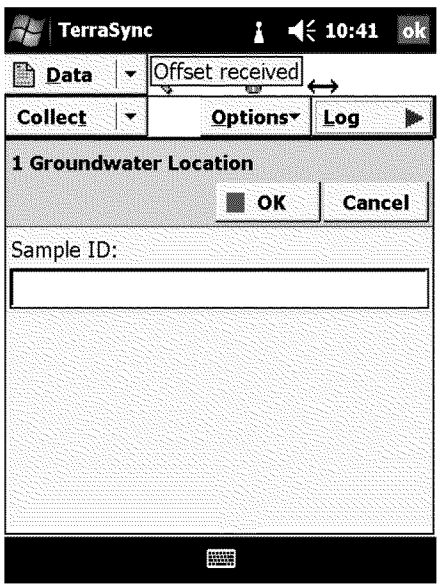
The LTI TruPulse 360 Laser RangeFinder is a measurement device that emits a laser to a distance of up to 1,000 meters and returns various information, such as vertical distance, horizontal distance, slope distance, inclination, azimuth, and height.

8.1 Enabling the Laser RangeFinder for Offset Point Collection

The Trimble units have been configured with the Laser setup and are ready to work. Laser #1 is setup for GPS units 1 & 2. Laser #2 is setup for GPS units 3 & 4. Before using the laser on your GPS device, you need to turn on Bluetooth on the Laser Rangefinder. To do this, look into the Laser eye piece and turn on the laser by clicking the **FIRE** button on top of the laser. Press the down arrow for four seconds. Press the down arrow once to get to the Blue Tooth option. Select **FIRE**. You have now enabled Blue Tooth on the laser. It should read **Bt_on**. Press **Fire** to return to main menu. Once the laser is enabled, it should be ready to work when you open a data file in the **Data** section of TerraSync.

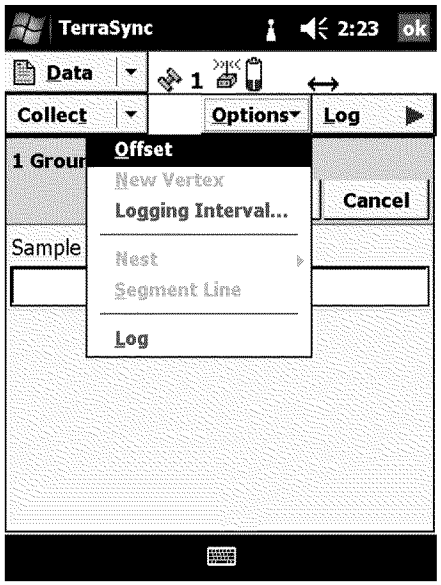
8.2 Using the Laser RangeFinder in TerraSync

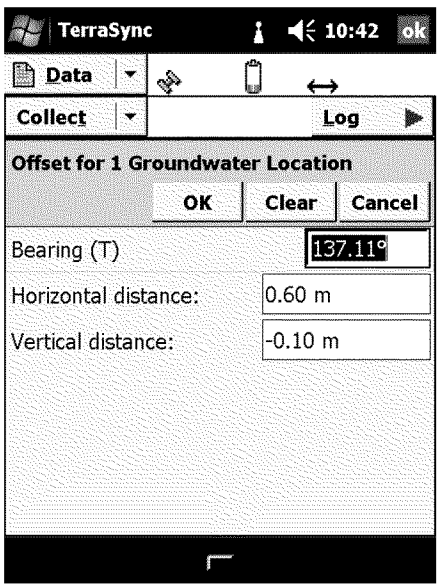
To use the laser once setup is complete, follow the same steps in data collection and begin logging points. After naming your sample ID, begin logging points. Aim the Laser RangeFinder at your target and push the **FIRE** button. You will be given notice that the offset has been received. Make sure that the **Offset received** notification appears. Sometimes your laser shot will not register and you will need to repeat your shot. When finished with the laser, it will turn off by itself.



REFER TO SECTION 8.3 TROUBLESHOOTING THE LASER RANGEFINDER IF YOU EXPERIENCE ANY DIFFICULTIES IN THE SETUP PROCESS.

To confirm the offset distance, you can tap the **Options** button and then click **Offset** to view the offset information that has been gathered.





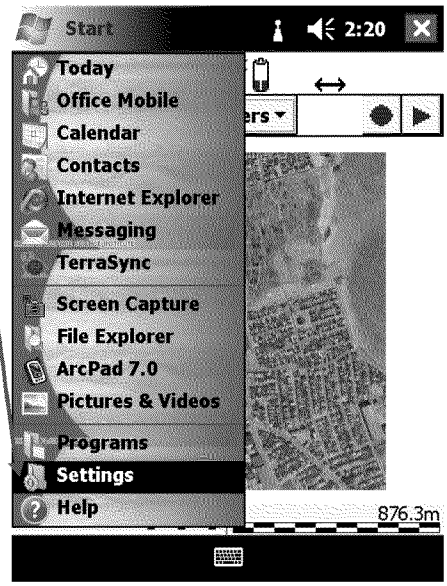
Your offset has been captured. Click **OK** and then click **OK** again to capture the offset point.

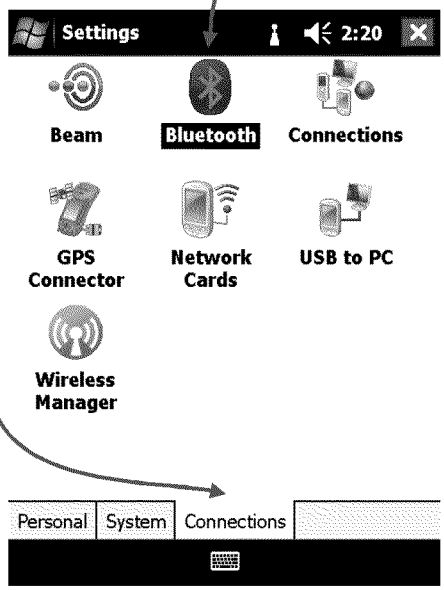
For additional setting and configuration options for the laser, refer to the TruPulse 200B/36B Cheat Notes card. This card can be found in the Laser Box.

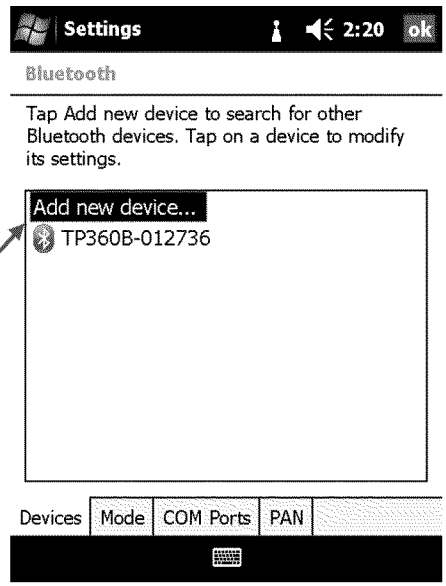
8.3 Troubleshooting the Laser RangeFinder

If enabling the laser did not work as described above, you will have to reestablish and reconfigure the Bluetooth connection to the Trimble unit. This is common so do not panic! At times, this process may seem to loop on itself. Follow these instructions and you will be ready to use the laser in very little time.

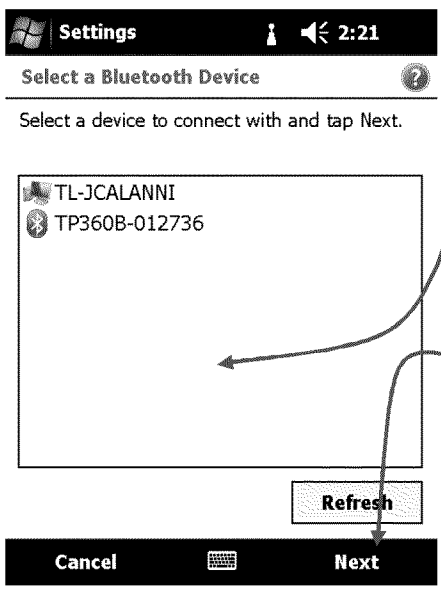
First ensure that the laser's Bluetooth is turned on.
To activate the laser on your GPS device, tap **Start** and select **Settings.** At the bottom of the **Settings** page, select the **Connections** tab, then tap the **Bluetooth** icon.



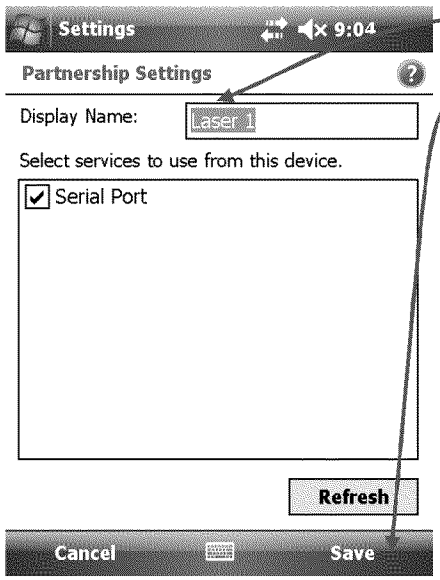




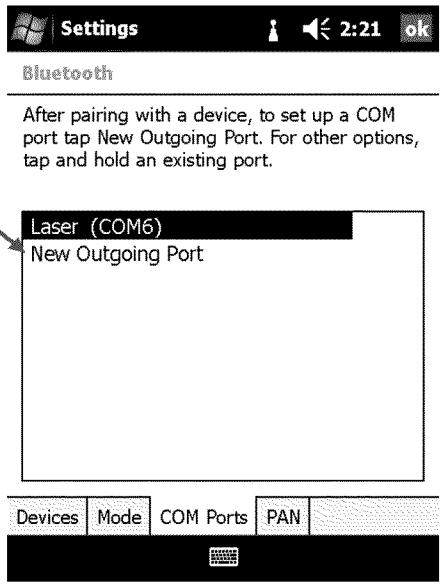
Choose **Add new device.**



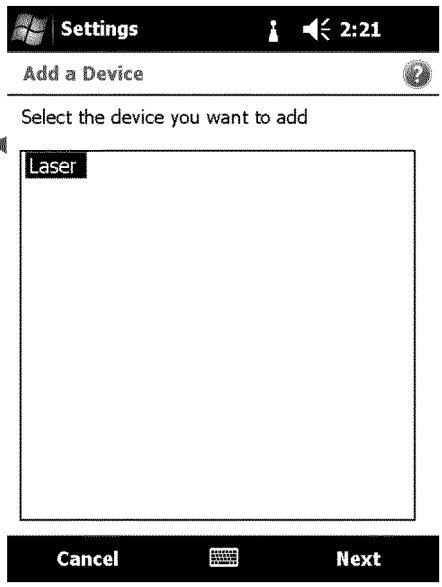
Select the Bluetooth Icon with label TP360B-012736. This number code is for Laser 1 (used with GPS unit 1 and 2); if using Laser 2 (for use with GPS units 3 and 4) the number code will be TP360B-017564. Tap **Next**. You will be prompted to enter the following pass code: 1111

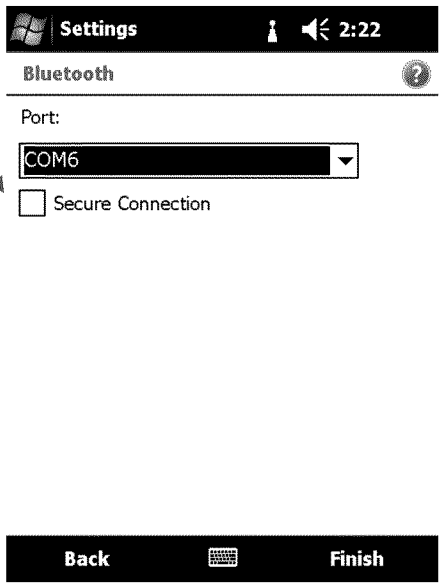


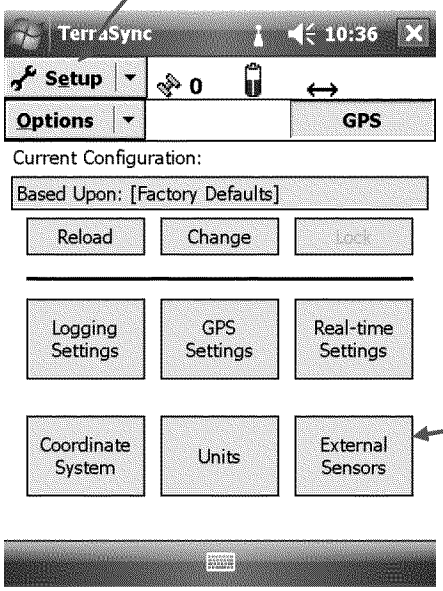
Check **Serial Port**, rename the **Display Name** "Laser," and click **Save** (left). In the next screen, choose **COM Ports** and select **New Outgoing Port**.



Select **Laser** and tap **Next**. On the next screen, choose a COM Port from the drop down. ***Do not check Secure Connection.*** Tap **Finish. Close this application**.

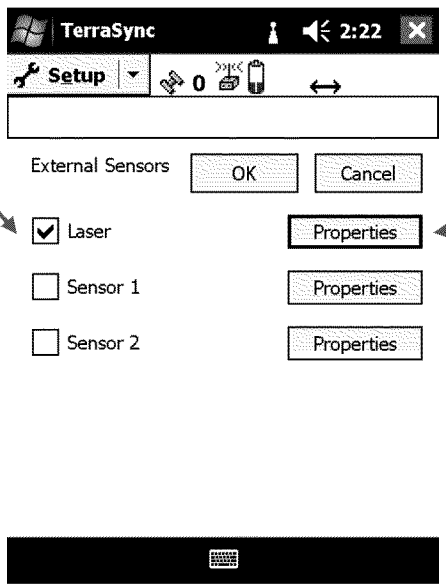




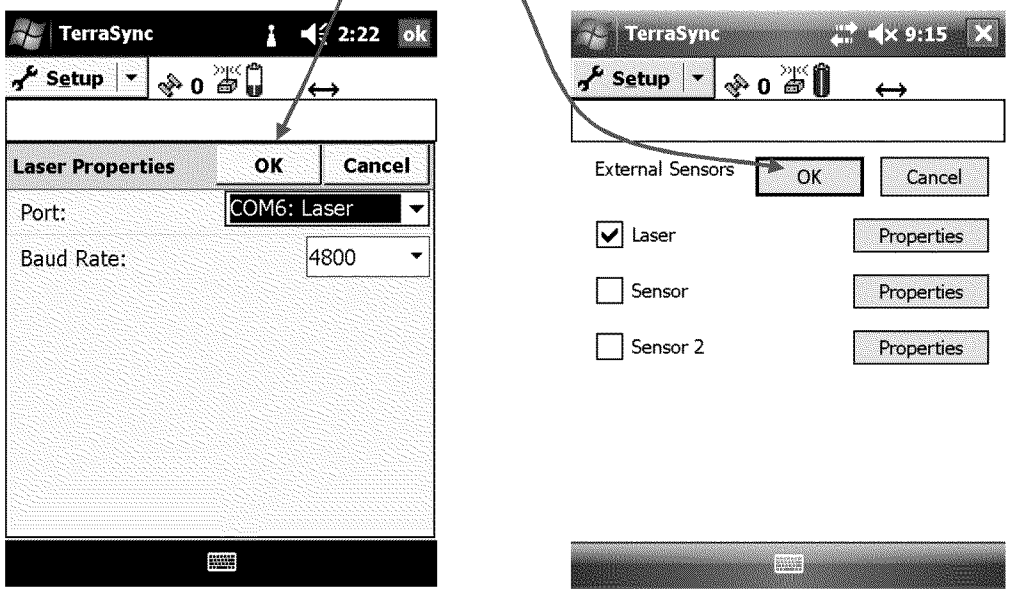


Back in TerraSync, tap the **Setup** menu and choose **External Sensors** to link to the Laser Rangefinder.

Check the box next to **Laser** to enable. Next tap the **Properties** box to setup the correct COM Port.



Tap the drop down box next to **Port** and choose the COM Port that you assigned to the **Laser**. Tap **OK** and then tap **OK** on the next screen. You are now ready to take offset points using the Laser Rangefinder.



9.0 TROUBLESHOOTING

Sometimes the units go into deep hibernation and will not turn on using the on/off button. To fix this, the power button and tiny grey reset button need to be depressed simultaneously. Use a pen to press the reset button (inset on the upper left of the Trimble keypad).

9.1 Contact numbers for assistance

Ryan Bahnfleth, ESAT 303.312.7723
Dan Michor, ESAT 303.312.7715
Trimble Support 1.800.728.5066

10.0 REFERENCES

Trimble Navigation Limited. Trimble Mapping and GIS Products: New Post-Processing Engine. 2009. Westminster, CO, USA.

Trimble Navigation Limited. TerraSync Software Reference Manual. 2008, Version 3.21, Revision A. Westminster, CO, USA.

Trimble Navigation Limited. GeoExplorer 2008 Series User Guide. 2008, Version 1.00, Revision B. Westminster, CO, USA.

Sample Custody and Labeling

APPROVED:

_____ _____
ESAT Region 8 QA Coordinator Date

_____ _____
ESAT Region 8 Team Manager Date

_____ _____
EPA Task Order Project Officer Date

_____ _____
ESAT Region 8 Task Lead Date

DCN: EP8-6-6513

This document has been prepared for the Environmental Protection Agency by the TechLaw, Inc. ESAT Region 8 Team and is intended to provide documentation of administrative, analytical and quality control procedures used in the daily performance of EPA and ESAT support services.

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to assist field personnel in developing proper sample custody and sample identification methods for the collection of environmental samples. This includes the use of chain of custody (COC) forms and labels for samples collected in the field. These procedures are critical in ensuring the integrity of environmental samples.

2.0 SCOPE AND APPLICABILITY

To ensure the integrity of a sample collected in the field or generated in a laboratory setting, documentation is needed to chronicle all sample handling for collection or creation through analysis and/or disposal. Any sample that is collected in the field or generated in a laboratory setting will require that records are kept as it transfers from various entities. This is the basis for generation of a COC. Uniquely, labeling samples with information, such as sample location, date, time, preservation method, and analytical requirements, keeps samples organized. A COC is initiated for each sample, either at the time of sample collection or generation or as part of preparation for a sampling event. This SOP will cover the best practices for sample custody and the method of COC and label generation.

3.0 SUMMARY OF METHOD

Once a sample is collected, several steps need to be taken to ensure the required information is collected and maintained as it is transferred from the point of collection to the laboratory. If sample nomenclature and location is known before a field event, a COC will be generated before deployment into the field. When generating the COC, it is important to know the analytical fate of samples required for each sample location (e.g. total recoverable metal, dissolved metals, etc.). This information can be found in the site-specific Sampling and Analysis Plan (SAP) and other sampling event planning documents. Some software programs (e.g. Scribe) that generate COCs also have the ability to generate labels. Scribe is the Laboratory Information Management System (LIMS) used by the lab. It is important to keep in mind that it is not mandatory to generate COCs and labels before a sampling event, but it is preferred. If it is not known where samples will be collected or the nomenclature of the sites is unclear, sample containers can be labeled with permanent marker with tape placed over it, and a blank COC can be filled out at the time of sample collection. Once the method of custody is established, a specific person, known as the sample custodian, is then responsible for maintaining the integrity of the samples as they move from and within various locations.

4.0 ACRONYMS AND DEFINITIONS

CLP Contract Lab Program
COC Chain of Custody
EPA United States Environmental Protection Agency
ERT Environmental Response Team
ID Identification
LIMS Laboratory Information Management System
QAPP Quality Assurance Project Plan
SAP Sampling and Analysis Plan
SOP Standard Operating Procedure

Chain of Custody (COC): A document used to chronologically track movement of samples between entities from collection to disposal.

Sampling and Analysis Plan (SAP): A site-specific document that describes the events to take place in the field.

Scribe: – A software tool developed by the United States Environmental Protections Agency (EPA) Environmental Response Team (ERT) to assist in the process of managing environmental data. Scribe captures sampling, observational, and monitoring field data.

Standard Operating Procedure (SOP): A set of written instructions that document a routine or repetitive activity followed by an organization (EPA, 2007).

5.0 HEALTH AND SAFETY

There are no specific health and safety hazards associated with sample custody and labeling, but these activities sometimes take place on-site during a sampling event. It is important for field personnel to familiarize themselves with the site-specific Health and Safety Plan before deployment to a site. In terms of personal interaction with the sample throughout the process of sample custody, there exists the possibility that the samples can leak. It is important to be aware of such hazards, especially when interacting with samples that are highly contaminated.

6.0 CAUTIONS

Samples sometimes require specific storage and maintenance, such as temperature preservation requirements. Proper storage of samples is critical in maintaining their integrity. Labeling is also critical in the process of sample custody. Samples usually are labeled with a series of letters and numbers that correspond to a site location, which sometimes are very similar to each other. Sample nomenclature will be designated in the approved SAP and will be followed in the field. Once a COC or label is generated, it is very important to have it reviewed for quality assurance purposes. Sample label and COC review is necessary to ensure that they match site documents.

7.0 INTERFERENCES

Once a COC and group of labels are reviewed and deployed, it is critical that the proper label ends up on the correct sample container. There will be more than one subsample collected at the majority of sampling locations in the region. This means that sample numbers can be very close in nomenclature, which puts more emphasis on attention to detail when labeling the sample containers. If the wrong label is attached to a sample, it may result in improper preservation, improper analysis, or rejection by the analytical laboratory.

8.0 PERSONNEL QUALIFICATIONS

It is critical that field personnel have proper clearance and health and safety training. Anyone who performs sample custody activities should also familiarize themselves the site-specific SAP and Quality Assurance Project Plan (QAPP), as well as with applicable SOPs: Surface Water Sampling SOP FLD 1.00, Groundwater Sampling SOP FLD 04.00, Soil Sampling SOP FLD 5.00, Pore Water Sampling SOP FLD 10.00, and Shallow Stream Sediment Sampling SOP FLD 06.00.

9.0 EQUIPMENT AND SUPPLIES

Below is a list of equipment and supplies required for COC activities (refer to the site specific SAP for additional items that maybe needed:

- Scribe software
- A SAP that details sample locations and analytical requirements
- Printer (that accepts corresponding labels)
- Blank COC pages in case of unexpected opportunistic sampling
- Permanent marker for preliminary labeling
- Clear tape for label protection from moisture
- Printable labels
- Field Logbook

10.0 STANDARDS AND REAGENTS

There are no standards or reagents associated with this SOP.

11.0 PROCEDURES

The following sections outline the general procedures for sample custody and labeling, filling out COCs with the proper information, and relinquishing samples. See Attachment A for an example of a blank COC and Attachment B for an example of a sample label.

11.1 Generating a Blank COC and Sample Labels

There are several types of data management software that can be used to generate COCs and labels. Scribe is used at the EPA Region 8 laboratory. Some training is required before an individual can use Scribe; however, once the basics of Scribe are understood, it can be used to generate COCs and labels for any type of sample or analysis. A COC that is generated prior to deployment should have the following information:

- Site Identification
- Analysis to be performed
- Preservation
- Tag Identification

The following information should not be filled out until sampling occurs:

- Date
- Time
- Sampler identification
- Comments describing anomalies

Labels can be produced with the same information found in the COCs.

11.2 Populating COC Fields and Affixing Labels

Sample containers should always be marked with a permanent marker with the site identification (ID), time of collection, analysis to be performed, date, and sampler initials prior to sample collection. Once samples have been collected, and a safe place to fill out COC and labels is established, field personnel should fill out the pre-populated COCs and labels with information such as date, time of collection, sampler initials, and comments. It is imperative that the information written on the sample container in permanent marker is the same information on the sample labels and the COC. The same information should also be recorded in a site-dedicated field logbook.

Once the labels have been verified to have the correct information, they should be affixed to the sample containers. Always be sure to double check that the proper label is placed on the corresponding sample container by cross-referencing it with the markings. Once the label is affixed to the sample container, place clear packing tape over the label and wrap completely around the container. This will prevent moisture from dissolving the label adhesive and blurring the writing. It also prevents holes, knicks, or tears from rendering the label unreadable.

11.3 Review/Custody Transfer

Once sample information is written on the COC and labels, and the label IDs have been verified against the permanent marker ID on the container, they are then ready for transfer of custody. Whether the samples are going to the EPA Region 8 lab or a Contract Lab Program (CLP) laboratory partner, the samples must be properly shipped at the required temperature (4°C for water and sediment samples) and done so in a way that containers are not compromised. In order to not compromise the integrity of the samples, the handler needs to make sure the cooler or other transporting vessel is not dropped, exposed to moisture or extreme weather, or in any other way disturbed. A signed copy of the COC intended for the receiving laboratory (samples IDs and event information should not be viewable to the lab) must be included in the shipping container. If samples are returning to the Region 8 Laboratory, they should be properly stored on ice in the field until delivered to the lab. To protect against sample contamination, place the ice in the coolers in plastic bags. When at the lab, samples should be placed in the walk-in coolers located in the sample receiving room. A signed copy of the COC is given to the sample receiving coordinator. In order to ensure samples are transferred to the correct party with the appropriate information and communication, a mutual signing of the COC by the sampler or transport agency and the sample coordinator can be arranged.

12.0 DATA RECORDS AND MANAGEMENT

As mentioned earlier, a COC should have information such as site ID, sample location, sample time, sample date, sampler initials, analytical requirements, sample matrix, preservative type, and a comments field. A sample label should have information such as sample location, time, date, matrix, preservative, and sampler initials. Any other field observations that require an explanation should be noted in the field forms or site-dedicated field notebook. Data such as sample ID, time, date, field parameters, (pH, temperature, conductivity, and dissolved oxygen) and sampler initials will eventually be entered into Scribe.

13.0 QUALITY CONTROL AND ASSURANCE

Proper sample custody and labeling requires a number of quality control and assurance steps. A COC generated in Scribe should always be crossed-checked by another person with the sample list found in the SAP. Completed COCs and labels should also be compared for accuracy before being relinquished to the receiving analytical laboratory. Any incorrect information on a COC or label may cause the lab to reject the shipment.

14.0 REFERENCES

EPA Guidance for Preparing Standard Operating Procedures, EPA QA/G-6, April 2007.

15.0 ATTACHMENTS

Attachment A: Example Chain of Custody Form

TechLaw
ESAT Region 8 Laboratory
16194 W 45th Drive
Golden, CO 80403
303.312.7047

US EPA CLP
Chain-of-Custody

Page 1 of 3

EVENT: 2011_eCOC Template

Sample #	Tag	Location	Sub Location	Sample Type	Collection	Matrix	Analyses	Preservation	Sample Date	Sample Time	Sampler	Remarks

Relinquished By (DATE):

Relinquished By:

Cooler Temp:_____
ICE: Y N
pH: Y N
Cust. Seals: Y N
COC/Labels Agree: Y N
Containers Intact: Y N

Received By (DATE/TIME):

Received By:

Attachment B: Example Sample Label

Sample # 082X-127 **Sampler:**
Tag: A
Date: Sample Time:
Location: Dup-05 Samp_Depth:
Analyses: Total Recoverable Metals
Preservation: TR_Plastic Baggie

General Field Sampling Protocols

APPROVED:

_____ _____
ESAT Region 8 QA Coordinator Date

_____ _____
ESAT Region 8 Team Manager Date

_____ _____
EPA Task Order Project Officer Date

_____ _____
ESAT Region 8 Task Lead Date

DCN: EP8-6-6513

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14.0 REFERENCES 9

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide general field sampling guidelines that will assist Environmental Protection Agency (EPA) and Environmental Services Assistance Team (ESAT) personnel in choosing sampling strategies, location, and frequency for proper assessment of site characteristics. This SOP is applicable to all field activities that involve sampling.

2.0 SCOPE AND APPLICABILITY

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent on site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

3.0 SUMMARY OF METHOD

Sampling is the selection of a representative portion of a larger population, area or body. Through examination of a sample, the characteristics of the larger entity from which the sample was drawn can be inferred. In this manner, sampling can be a valuable tool for determining the presence, type, and extent of contamination by hazardous substances in the environment. The sampling design is a fundamental part of data collection for scientifically based decision making. A well-developed sampling design plays a critical role in ensuring that data are sufficient to draw the conclusions needed. The goals of a sampling design can vary widely. Typical objectives of a sampling design for environmental data collection are:

- To support a decision about whether contamination levels exceed a threshold of unacceptable risk
- To determine whether certain characteristics of two populations differ by some amount
- To estimate the mean characteristics of a population or the proportion of a population that has certain characteristics of interest
- To identify the location of “hot spots” (areas having high levels of contamination) or plume delineation
- To characterize the nature and extent of contamination at a site
- To monitor trends in environmental conditions or indicators of health

A well-planned sampling design is intended to ensure that resulting data are adequately representative of the target population and defensible for their intended use. Representativeness may be considered as the measure of the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Throughout the sampling design process, the efficient use of time, money, and human resources are critical considerations. A good design should meet the needs of the study with a minimum expenditure of resources. If resources

4.0 ACRONYMS AND DEFINITIONS

EPA United States Environmental Protection Agency
ESAT Environmental Services Assistance Team
DOT Department of Transportation

HAZWOPER Hazardous Waste Operations and Emergency Response
IATA International Air Transport Association
MI Multi-increment
OSHA Occupational Safety and Health Administration
QA Quality Assurance
QAPP Quality Assurance Project Plan
QC Quality Control
SAP Sampling and Analysis Plan
SOP Standard Operating Procedure

Occupational Safety and Health Administration (OSHA): A regulatory agency that governs health and safety standards in the United States.

Standard Operating Procedure (SOP): A set of written instructions that document a routine or repetitive activity followed by an organization (EPA, 2007).

Quality Assurance Project Plan (QAPP): A site-specific document that specifies quality assurance activities and data quality objectives.

5.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. Environmental Protection Agency (EPA), Occupational Safety and Health Administration (OSHA) and corporate health and safety procedures. Always review the site Health and Safety Plan (HASP) before beginning work at any site.

6.0 CAUTIONS

In general, health and safety of field team members and sample/data integrity are the two main concerns during a field sampling event. Field personnel must understand sampling procedures and be familiar with health and safety protocols before deployment to a site. Always consult the HASP before entering a site.

7.0 INTERFERENCES

The nature of the object or materials being sampled may be challenging to characterize. If a material is homogeneous, it will generally have a uniform composition throughout. In this case, any sample increment can be considered representative of the material. On the other hand, heterogeneous samples present problems to the sampler because of spatial and temporal changes in the material. Samples of hazardous materials may pose a safety threat to both field and laboratory personnel. Proper health and safety precautions should be implemented when handling this type of sample. Environmental conditions, weather conditions, or non-target chemicals may cause problems and/or interferences when performing sampling activities or when sampling for a specific parameter. Refer to the specific SOPs for sampling techniques.

8.0 PERSONNEL QUALIFICATIONS

All personnel who participate in field activities are required to obtain clearance in three mandatory health and safety programs: medical monitoring, respirator fit testing, and OSHA Hazardous Waste Operations

and Emergency Response (HAZWOPER) 40-hour training. In addition, any personnel who will participate in sampling activities must read, understand, and sign the site-specific HASP and associated Sampling and Analysis Plan/Quality Assurance Project Plan (SAP/QAPP).

9.0 EQUIPMENT AND SUPPLIES

The equipment required to collect samples must be determined on a site-specific basis. Due to the wide variety of sampling equipment available, refer to the specific SOPs for sampling techniques which include lists of the equipment required for sampling.

10.0 STANDARDS AND REAGENTS

Reagents may be utilized for preservation of samples and for decontamination of sampling equipment. The preservatives required are specified by the analysis to be performed. Decontamination solutions are specified in the Sampling Equipment Decontamination SOP FLD 02.00.

11.0 PROCEDURES

11.1 Types of Samples

In relation to the media to be sampled, two basic types of samples can be considered: the environmental sample and the hazardous sample.

Environmental samples are those collected from streams, ponds, lakes, wells, and are off-site samples that are not expected to be contaminated with high levels of hazardous materials. They usually do not require the special handling procedures typically used for concentrated wastes. However, in certain instances, environmental samples can contain elevated concentrations of pollutants and in such cases would have to be handled as hazardous samples.

Hazardous or concentrated samples are those collected from drums, tanks, lagoons, pits, waste piles, fresh spills, or areas previously identified as contaminated, and require special handling procedures because of their potential toxicity or hazard. These samples can be further subdivided based on their degree of hazard; however, care should be taken when handling and shipping any wastes believed to be concentrated regardless of the degree. The importance of making the distinction between environmental and hazardous samples is two-fold:

1. Personnel safety requirements: Any sample thought to contain enough hazardous materials to pose a safety threat should be designated as hazardous and handled in a manner which ensures the safety of both field and laboratory personnel. Personnel handling potentially hazardous substances should always wear proper Personal Protective Equipment.

2. Transportation requirements: Hazardous samples must be packaged, labeled, and shipped according to the International Air Transport Association (IATA) Dangerous Goods Regulations or Department of Transportation (DOT) regulations and U.S. EPA guidelines.

11.2 Sample Collection Techniques

In general, two basic types of sample collection techniques are recognized, both of which can be used for either environmental or hazardous samples.

Grab (Discrete) Samples
A grab sample is defined as a discrete aliquot representative of a specific location at a given point in time. The sample is collected all at once at one particular point in the sample medium. The representativeness of such samples is defined by the nature of the materials being sampled. In general, as sources vary over time and distance, the representativeness of grab samples will decrease.

Composite (Multi-Increment) Samples
Multi-increment (MI) or composite sampling is a structured sampling protocol that reduces data variability and increases sample representativeness. The objective of MI sampling is to obtain a single sample for analysis that has a mean analyte concentration representative of the decision unit. The decision unit size is site-specific and represents the smallest area on which to base a decision or conclusion. Samples are collected from multiple locations within the decision unit and composited so the samples are spatially representative of the decision unit. The decision unit must be defined so that the results are relevant to explicitly articulated sampling objectives. Note that establishment of decision units is necessary to develop any effective sampling approach, whether using MI or discrete sampling.

The MI sampling strategy improves the reliability and defensibility of sampling data by reducing their variability compared to conventional discrete sampling strategies. The data distribution for MI replicate samples tends to be normally distributed, as contrasted to the positively skewed distribution seen with discrete samples. Fewer non-detect results can be expected using MI, thus mitigating problems caused by using censored data sets and lessening the chance of missing significant contamination. In addition, levels of statistical confidence and decision uncertainty that would require a large number

11.3 Types of Sampling Strategies

It is important to select an appropriate sampling approach for accurate characterization of site conditions. Prior to undertaking any sampling program, it is necessary to establish appropriate measurement and system Data Quality Objectives. Refer to the U.S. Environmental Protection Agency (EPA) Soil Sampling Quality Assurance User's Guide (listed in Section 14.0 References) for guidance in establishing Data Quality Objectives, statistical sampling methodologies and protocols for each of the sampling approaches. Each approach is defined below.

Judgmental or Biased Sampling
Judgmental or Biased sampling is used primarily for documenting an observed release to the groundwater, surface water, air or soil exposure pathways. This form of sampling is based on the subjective selection of sampling locations where contamination is most likely to occur. Locations are based on relative historical site information and on-site investigation (site walk-over) where contamination is most likely to occur.

There is no randomization associated with this sampling approach because samples are primarily collected at areas of suspected highest contaminant concentrations. Any statistical calculations based on the results of this sampling technique will be biased.

Random Sampling
Random sampling, used for the characterization of a heterogeneous non-stratified waste, involves arbitrary collection of samples within a defined area. This method is most effective and accurate if the chemical heterogeneity of the waste remains constant from batch to batch. The easiest method for Random Sampling is to divide the area for sampling into an imaginary grid, assign a series of numbers to the units of the grid, and select the numbers or units to be sampled through the use of a random-numbers table which can be found in the text of any basic statistics book. Note that haphazardly selecting sample numbers or units is not a suitable substitute for a randomly selected sample.

Stratified Random Sampling
Stratified random sampling, used for the characterization of a heterogeneous stratified waste, involves arbitrary collection of samples within a defined area and strata. This method is most effective and accurate if the chemical heterogeneity of the waste remains constant from batch to batch. The easiest method for stratified random sampling is to divide the area for sampling into an imaginary grid, assign a series of numbers to the units of the grid, and select the numbers or units to be sampled through the use of a random-numbers table which can be found in the text of any basic statistics book. A random sample is then collected from each strata at the selected numbers or units on the grid. Note that haphazardly selecting sample numbers or units is not a suitable substitute for a randomly selected sample.

Systematic Grid Sampling
Systematic grid sampling involves dividing the area of concern into smaller sampling areas using a square or triangular grid. Samples are then collected from the intersection of the grid lines or nodes. The origin and direction for placement of the grid should be selected by using an initial random point. The distance between nodes is dependent upon the size of the site or area of concern and the number of samples to be collected. Generally, a larger distance is used for a large area of concern.

Systematic Random Sampling
Systematic random sampling involves dividing the area of concern into smaller sampling areas. Samples are collected within each individual grid cell using random selection procedures.

Search Sampling
Search sampling utilizes a systematic grid or systematic random sampling approach to define areas where contaminants exceed clean-up criteria. The distance between the grid lines and number of samples to be collected are dependent upon the acceptable level of error (i.e., the chance of missing a hot spot). This sampling approach requires that assumptions be made regarding the size, shape, and depth of hot spots.

Transect Sampling
Transect sampling involves establishing one or more transect lines, parallel or non-parallel, across the area of concern. If the lines are parallel, this sampling approach is similar to systematic grid sampling. The advantage of transect sampling over systematic grid sampling is

the relative ease of establishing and relocation transect lines versus an entire grid. Samples are collected at regular intervals along the transect line at the surface and/or at a specified depth(s). The distance between the sample locations is determined by the length of the line and the number of samples to be collected.

11.4 Quality Assurance Project Plans (QAPP)

A Quality Assurance Project Plan (EPA, 2006) is required when it becomes evident that a field investigation is necessary. It should be initiated in conjunction with, or immediately following, notification of the field investigation. This plan should be clear and concise and should detail the following basic components, with regard to sampling activities:

- Objective and purpose of the investigation
- Basis upon which data will be evaluated
- Information known about the site including location, type and size of the facility, and length of operations/abandonment
- Type and volume of contaminated material, contaminants of concern (including concentration), and basis of the information/data
- Technical approach including media/matrix to be sampled, sampling equipment to be used, sample equipment decontamination (if necessary), sampling design and rationale, and SOPs or description of the procedure to be implemented
- Project management and reporting, schedule, project organization and responsibilities, manpower and cost projections, and required deliverables
- QA objectives and protocols including tables summarizing field sampling and QA/QC analysis and objectives

Note that this list of QAPP components is not all-inclusive and that additional element(s) may be added or altered depending on the specific requirements of the field investigation. It should also be recognized that although a detailed QAPP is quite important, it may be impractical in some instances. Emergency responses and accidental spills are prime examples of such instances where time might prohibit the development of site-specific QAPPs prior to field activities. In such cases, investigators would have to rely on general guidelines and personal judgment, and the sampling or response plans might simply be a strategy based on preliminary information and finalized on site. In any event, a plan of action should be developed, no matter how concise or informal, to aid investigators in maintaining a logical and consistent order to the implementation of their task.

11.5 Legal Implications

The data derived from sampling activities are often introduced as critical evidence during litigation of a hazardous waste site cleanup. Legal issues in which sampling data are important may include cleanup cost recovery, identification of pollution sources and responsible parties, and technical validation of remedial design methodologies. Because of the potential for involvement in legal actions, strict adherence to technical and administrative SOPs is essential during both the development and implementation of sampling activities.

Technically valid sampling begins with thorough planning and continues through the sample collection and analytical procedures. Administrative requirements involve thorough, accurate documentation of all sampling activities. Documentation requirements include maintenance of a chain of custody, as well as accurate records of field activities and analytical instructions. Failure to observe these procedures fully and consistently may result in data that are questionable, invalid and non-defensible in court, and the consequent loss of enforcement proceedings.

12.0 DATA RECORDS AND MANAGEMENT

There are many data parameters and custody records that require attention to detail. Refer to the specific SOPs for data management activities that are associated with sampling techniques.

13.0 QUALITY CONTROL/QUALITY ASSURANCE (QC/QA)

Refer to the specific SOPs for the type and frequency of QA/QC samples to be analyzed, the acceptance criteria for the QA/QC samples, and any other QA/QC activities which are associated with sampling techniques.

14.0 REFERENCES

EPA Guidance for Preparing Standard Operating Procedures, EPA QA/G-6. April 2007.
EPA Guidance on Systematic Planning using the Data Quality Objectives Process (QA/G-4). February 2006.

EPA Guidance on Choosing a Sampling Design for Environmental Data Collection (QA/G-5S). December, 2002

ANALYTICAL DETERMINATION OF TRACE METALS IN SOIL AND SEDIMENT BY FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY

APPROVED:

_____ _____
ESAT Region 8 QA Coordinator Date

_____ _____
ESAT Region 8 Team Manager Date

_____ _____
EPA Task Order Project Officer Date

_____ _____
ESAT Region 8 Task Lead Date

DCN: EP8-6-6513

This document has been prepared for the Environmental Protection Agency by the TechLaw, Inc. ESAT Region 8 Team and is intended to provide documentation of administrative, analytical and quality control procedures used in the daily performance of EPA and ESAT support services.

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1.0 SCOPE AND APPLICABILITY

This standard operating procedure (SOP) provides procedures for the determination of elements in solid matrix samples. The X-Ray Fluorescence (XRF) provides analytical results for metals using a portable, hand-held instrument. The Innov-X Systems XRF Analyzer (XRFA) will be used as a field screening method to analyze in-situ (directly on the ground), bagged or prepared soil samples. The method is useful in that it incorporates a large number of screening-level measurements that provide better characterization than a small number of laboratory-based measurements. The method will be used as a screening tool along with confirmatory analysis using Environmental Protection Agency (EPA) approved methods.

The method will be used in a number of field exercises including identification of hot spots or contaminated sites, site investigation/assessment and confirmation of remediation applications. The method's strength is the ability to provide immediate results to allow quick decisions to be made in the field. The method's weakness is detection limits above the toxicity characteristic regulatory level of some Resource Conservation and Recovery Act (RCRA) analytes. The detection limits depend on the analyte of interest, the strength of the excitation source, count times used, matrix effects and inter-elemental spectral interferences.

2.0 SUMMARY OF METHOD

XRF is a nondestructive qualitative and quantitative analytical technique used to determine the chemical composition of a sample. An element is identified by exposing a substance to high energy photons emitted from an x-ray tube. The incident radiation causes electrons to be ejected from the atomic shells of any elements present in the sample and as a result energy is released (fluoresced). The fluorescent energy is detected by the XRF analyzer as a characteristic x-ray spectra. The x-ray spectra emitted is characteristic for each element and is the qualitative analysis. The number of counts at a characteristic energy level per unit time is representative of the element concentration in a sample and is the basis for the quantitative analysis.

The hand-held, battery operated field portable XRFA is factory calibrated and generally does not require recalibration. Certified standards are used as the continuing calibration standards to check the initial factory calibration. Discrete samples are collected in suitable sampling containers and are homogenized before on-site analysis. The samples are crushed and sieved and put in sample cups for XRFA analysis.

3.0 ACRONYMS

As	Arsenic
°C	Degrees Celsius
Co	Cobalt
Cr	Chromium
Cu	Copper
CVC	Calibration Verification Check
DQO	Data Quality Objective
EPA	US Environmental Protection Agency
Fe	Iron
FP	Fundamental Parameter
ICP	Inductively Coupled Plasma
Hg	Mercury

MDL	Method Detection Limit
mR/hr	MilliRoentgen Per Hour
NIST	National Institute of Standards and Technology
PPE	Personal Protective Equipment
ppm	Parts Per Million
RCRA	Resource Conservation and Recovery Act
RSD	Relative Standard Deviation
SRM	Standard Reference Material
SSCS	Site Specific Calibration Standard
TBD	To Be Determined
V	Vanadium
XRF	X-Ray Fluorescence
XRFA	Innov-X Systems XRF Analyzer

4.0 HEALTH, SAFETY AND CAUTIONS

The XRFA is a safe instrument when used according to th e manufacturer's recommended safety procedures as detailed in the user manual. According to the manufacturer, radiation levels during testing were <0.1 mR/hr on all surfaces of the analyzer except at or near the exit port for the radiation. Therefore, if the operator follows the SOP and XRFA user manual, they will not obtain any detectable radiation dose above naturally occurring background radiation.

Proper use of the instrument includes never pointing the instrument at another person, pointing the instrument into the air to perform a test and never holding a sample in one's hand to perform the test.

The XRFA should be stored in a locked case or locked ca binets when not in use. The location of storage and use should be of restricted access to limit potential exposure to ionizing radiation.

Operators should minimize the time around the energized instrument, maximize the distance from the instrument window and shoot into high density materials whenever possible. Keep one's hand away from the source-end of the XRFA when the instrument is ionizing the sample. Always be aware of the instrument's radiation source and direction of beam of x-rays.

Each analyst should wear appropriate personal protective equipment (PPE), including gloves and safety glasses.

The XRFA should be transported in the waterproof, drop proof carrying case, equipped with padlocks and a lead plate for the source window. There are no travel restrictions regarding bridges, tunnels or planes. There may be disclosure and/or licensing requirements if the XRFA is taken across state or national boundaries. Check with appropriate agencies for these details.

5.0 INTERFERENCES

5.1 Method Error

Total method error for XRF analysis is defined as the square root of the sum of squares of both instrument precision and user- or application-related error. Generally, instrument precision is the least significant source of error in XRF analysis. User- or application-related error is generally

more significant and varies with each site and method used. Some sources of interference can be minimized or controlled by the instrument operator, but others cannot. Common sources of user- or application-related error are discussed below.

5.2 Physical Matrix Effects

This form of interference results from variations in the physical character of the sample.

These variations may include such parameters as particle size, uniformity, homogeneity, and surface condition.

Note: For example, if any analyte exists in the form of very fine particles in a coarser-grained matrix, the analyte's concentration measured by the XRF will vary depending on how fine particles are distributed within the coarser-grained matrix. If the fine particles "settle" to the bottom of the sample cup, the measurement will be higher than if the fine particles are mixed in well in the cup.

One way to reduce such error is to grind and sieve all soil samples to a uniform particle size, thus reducing sample to sample particle size variability.

Homogeneity is always a concern when dealing with soil samples. Every effort should be made to thoroughly mix and homogenize soil samples before analysis.

5.3 Moisture Content

Moisture in the sample may affect the accuracy of analysis of soil and sediment sample analyses.

When the moisture content is between 5 and 20 percent, the overall error from moisture may be minimal. However, moisture content may be a major source of error when analyzing samples of surface soil or sediment that are saturated with water.

This error can be minimized by drying the samples in a convection oven.

5.4 Positioning of Samples for Analysis

Inconsistent positioning of samples in front of the probe window is a potential source of error because the x-ray signal decreases as the distance from the radioactive source increases.

This error is minimized by maintaining the same distance between the window and each sample. For the best results, the window of the probe should be in direct contact with the sample, which means that the sample should be flat and smooth to provide a good contact surface.

5.5 Chemical Matrix Effects

This effect results from differences in the concentrations of interfering elements. These effects occur as either spectral interferences (peak overlaps) or as x-ray absorption and enhancement phenomena. Both effects are common in soils contaminated with heavy metals.

Note: As examples of absorption and enhancement effects; iron (Fe) tends to absorb copper (Cu) x-rays, reducing the intensity of the Cu measured by the detector, while chromium (Cr) will be enhanced at the expense of Fe because the absorption edge of Cr is slightly lower in energy than the fluorescent peak of Fe.

The effects can be corrected mathematically through the use of Fundamental Parameter (FP) coefficients.

The purpose of XRF analysis with Fundamental Parameters (FP) is to convert elemental peak intensities to elemental concentrations and/or film thicknesses.

This is achieved though a calibration step, where the XRF response function (related to parameters that are independent of the sample matrix) for each element is measured using a known standard of some kind. ESAT will never calibrate the XRF, as it will always be sent back to the manufacturer for such maintenance

The effects also can be compensated for using Site Specific Calibration Standards (SSCS), which contain all the elements present on site that can interfere with one another.

5.6 Spectrum Overlap

When present in a sample, certain x-ray lines from different elements can be very close in energy and, therefore, can cause interference by producing a severely overlapped spectrum.

The degree to which a detector can resolve the two different peaks depends on the energy resolution of the detector. If the energy difference between the two peaks in electron volts is less than the resolution of the detector in electron volts, then the detector will not be able to fully resolve the peaks.

The most common spectrum overlaps involve the K line of element Z-1 with the K line of element Z. This is called the Ka/Kb interference. For example, large amounts of vanadium (V) and Fe will interfere with the quantization of Cr or Cobalt (Co) respectively. The presence of Fe is a frequent problem because it is often found in soils at tens of thousands of parts per million (ppm).

5.7 Ambient Temperature Changes

Ambient temperature changes affect the gain of the amplifiers, producing instrument drift. The instrument should be recalibrated when large changes in ambient temperature are noted.

6.0 PERSONNEL QUALIFICATION

This test is restricted to use by or under the supervision of analysts experienced in the use of the XRFA. Each analyst must demonstrate the ability to generate acceptable results with this test method before approved to operate the instrument.

7.0 EQUIPMENT AND SUPPLIES

Innov-X-Systems XRF Analyzer, model Alpha 4000 S
Battery Charger
Extra Battery
Polyethylene sample cups, Chemplex Cat. No. 1330 30.7mm diameter x 23.5 mm, or equivalent
X-ray window film, Chemplex Cat. No. 425, polypropylene, or equivalent
#60 stainless steel sieve, top and bottom
Mortar and pestle
Grinders
Trowels for collecting and smoothing soil
Plastic baggies for collection and homogenization of soil samples
Drying oven
Kim wipes
Large squirt bottle with DI water
Permanent ink markers
Mallet or rock hammer
Beaker(s)
Logbooks, project and instrument

8.0 REAGENTS AND STANDARDS

Standards are specific for the site and the projects data quality objective's (DQOs). The XRFA is self-calibrating. However, standard reference materials (SRMs) should be incorporated into all projects to assure the self-calibration is operating properly. National Institute of Standards and Technology (NIST) SRM 2710, Montana Soil, is a good selection for an SRM and will be utilized for this method. SSCS may be used for better accuracy if required by the site DQOs.

8.1 Site-Specific Calibration Standards

The SSCS must be representative of the matrix to be analyzed by the XRFA. These samples must be well homogenized. A minimum of ten samples spanning the concentration ranges of the analytes of interest and of the interfering elements must be obtained from the site.

Each sample should be oven-dried for 2 to 4 hours at a temperature of less than 150 °C depending on DQOs. If mercury (Hg) or arsenic (As) are analytes of interest, samples should be dried at 60 °C for a minimum of 2 hours. When the sample is dry, all large, organic debris and non-representative material should be removed. The sample should be ground with a mortar and pestle and passed through a #60-mesh sieve.

The sample should be homogenized well. Approximately 5 grams of the sample should then be removed and placed in a sample cup for XRF analysis. The rest of the prepared sample is analyzed by Inductively Coupled Plasma (ICP). The method used for confirmatory analysis should meet the DQOs of the project.

Site Specific Calibration Standard (SSCSs) are used during a survey to predict the way the XRF will perform at a certain site. Data from the SSCSs are used to generate a statistical based curve in order to establish Action Levels and are not necessarily used during in situ analysis. Therefore, the calibration standard is not actually calibrating the XRF, but instead providing a

statistical correlation or a regression analysis.

8.2 Method Blank

The method blank consists of silica sand or lithium carbonate and must be free of any analytes a concentrations above the method detection limits (MDLs).

This method blank undergoes the same preparation procedures as the samples, and is used to monitor for cross-contamination and laboratory-induced contaminants or interferences.

8.3 Instrument Blank Standard

The blank standard consists of silicon dioxide, polytetrafluroethylene, a quartz block, “clean” sand, or lithium carbonate and must be free of analytes at concentrations above the method detection limits.

The instrument blank verifies that no contaminant of interest exists in the spectrometer or in on the probe window.

8.4 Standard Reference Materials

SRMs are standards containing certified amounts of metals in soil or sediment.

These standards are used for accuracy and performance checks of XRF analyses.

SRMs can be obtained from the NIST. NIST SRM 2710, Montana Soil will be used to verify the performance of the XRFA. The certified values for NIST 2710 can be found in Attachment I. NIST 2709 is used to establish the MDLs of the instrument and in order to do that it must be run a minimum of 7 times.

8.5 Second Source Standard

Solid matrix standards of known concentration that may be used for second source verification.

RTC certified XRF-408 standard will be used for second source verification. The certified values for RTC XRF-408 can be found in Attachment II.

9.0 PROCEDURE

9.1 Instrument Setup and Calibration

1. Prior to beginning a test, be certain the battery pack has sufficient charge. Always carry a spare battery pack. Place battery into analyzer.

2. Power on the XRFA main power (on/off switch located on back of analyzer). The green LED light next to the button will illuminate when the analyzer is on.

3. Power on the iPAQ (button located in upper right hand corner of iPAQ).

4. Select Innov-X from start menu located in upper left corner of iPAQ screen.

5. Read radiation safety notice and press start.

6. Select soil mode (XRFA undergoes 60 second hardware initialization).

7. From the Bluetooth Browser screen, select ok and proceed with standardization sequence.

8. Attach standardization clip or coupon (for use with test stand) on the nose of the analyzer and select standardization button on the screen.

   8a. Once standardized, remove clip or coupon.

   8b. Standardization must be performed any time the analyzer is initiated or restarted and should be repeated if the instrument is operating more than 4 hours.

   8c. Standardization is initiated from the Analysis Screen of any mode.

   8d. Record the energy resolution into the XRFA log book and select ok.

9. Select lock icon in the lower right hand corner of iPAQ screen to release trigger lock.

   9a. Test the required calibration check standard as required for the site and the project DQO's. This will be the NIST SRM 2710 or the SSCS as discussed in Section 6.0. Elemental concentrations should be within 20% of the standard value for all required analytes. The quality control requirements for the standard are detailed in Section 8.2. The calibration check standard values are reported in Appendixes I and II.

   9b. Analyze instrument calibration check blank labeled silicon dioxide. The instrument check blank confirms there is no contamination associated with the analyzer. The quality control requirements for the instrument blank are discussed in Section 8.3

   9c. Analyze method blank. This is only required if laboratory samples are prepared. This is used to verify that cross contamination has not occurred during the sample preparation process. The quality control requirements for the method blank are discussed in Section 8.4.

9.2 In-situ Sample Analysis

1. The detailed procedure will vary according to site specific plans, so the following is a general guideline. Consult the specific project SAP/QAPP for specific sampling details.

2. Select surface to be analyzed. Selected spot should not be saturated with water.

3. Clear any surface debris or vegetation. Level and flatten the exposed surface.

   3a. Hold the XRFA in one hand or place in analyzer holder. Do not place hand on the end of the plate of the instrument while measuring.

   3b. Place the XRFA on the polypropylene film on the ground of the sampling site and pull the trigger. The red light in the instrument will flash as long as the instrument is testing. The instrument is pre-programmed for the length of the test, typically 1-2 minutes. Pulling the trigger a second time will stop the test. All data will be stored in a data base of the PDA. After each test, the information (sampling location, operator, sample ID, etc) should be input into the data base for that sample.

9.3 Prepared Sample Analysis

1. The detailed procedure will vary according to site-specific plans, so the following is a general guideline. Consult the specific project SAP/QAPP for specific sampling details.

2. Select location to be analyzed. The sample location should have no greater than 25% moisture. (Rule of thumb, if sample remains clumped together after being squeezed by hand, sample needs to be dried.)

   2a. Remove any debris and other foreign objects from the surface so that the natural surface is exposed.

   2b. Using a sampling spade or trowel, take a vertical slice and place in a clean pail. Mix the sample by stirring and rotating the pail at a 45° degree angle. Don't shake the pail as this will stratify the sample by weight.

   2c. Collect a sample from a 4x4 inch square that is approximately 1/2 inch deep. Collect at least 100 grams of sample and place in a plastic baggie or plastic bucket and mix thoroughly. The sample will be homogenized, dried and ground before analysis. The sample can be homogenized before or after drying.

   2d. Dry the sample. The drying method will depend upon the project and may include drying for 2 to 4 hours in a convection oven at a temperature not greater than 150 °C. If As or Hg are required, drying at a temperature of 60°C for a minimum of 2 hours is recommended (until dry).

   2e. Sieve the sample using a clean #60 mesh screen until the required fraction is passed. Grinding is not recommended because it can alter the sample and negatively affect the data. Instead, large clumps should be broken up and the entire collected sample should be sieved. The sieving will allow adequate homogenization.

2f. Fill the XRF sample cup. Place a circle of 0.24 mil polypropylene film on top of the XRF cup. The top is the end of the cup with the indented ring. Secure the film with the collar. The film window should be taught and smooth. Tamp the sample into the cup and fasten the cap.

2g. Document the sampling identification on the XRF sample cup using a permanent ink marker. Set-up the XRFA using the testing platform. Samples are now ready for analysis.

2h. Pull the trigger on the XRFA. The red light in the instrument will flash as long as the instrument is testing. The instrument is pre-programmed for the length of the test, typically 1-2 minutes. Pulling the trigger a second time will stop the test. All data will be stored in a data base of the PDA. After each test, the information (sampling location, operator, sample ID, etc) should be input into the data base for that sample.

9.4 Laboratory Preparation and Analysis

1. The detailed procedure will vary according to site specific plans, so the following is a general guideline. Consult the specific project SAP/QAPP for specific sampling details.

2. The sample should be homogenized, dried, and ground before analysis. The sample can be homogenized before or after drying.

3. If the sample is homogenized before drying, it should be thoroughly mixed in a beaker or similar container, or if the sample is moist and has a high clay content, it can be kneaded in a plastic bag.

4. If the sample is homogenized after drying, spread out sample in shallow drying pan, break up any clumps with stainless steel spatula, air dry overnight, or oven dry for 2 to 4 hours in a convection oven at a temperature not greater than 150 °C. If As or Hg are required, dry at 60 °C for a minimum of 2 to 4 hours (until dry).

5. Sub-sample, grind, and sieve through a #60 mesh screen until enough sample has been collected to fill a XRF sample cup.

   5a. Fill the XRF sample cup. Place a circle of 0.24 mil polypropylene film on top of the XRF cup. The top is the end of the cup with the indented ring.

   5b. Secure the film with the collar. The film window should be taught and smooth.

   5c. Tap the sample into the cup and fasten the cap.

   5d. Document the sampling identification on the XRF sample cup using a

permanent ink marker.

5e. Set-up the XRFA using the testing platform. Samples are now ready for analysis.

5f. Pull the trigger on the XRFA. The red light in the instrument will flash as long as the instrument is testing. The instrument is pre-programmed for the length of the test, typically 1-2 minutes. Pulling the trigger a second time will stop the test.

6. All data will be stored in a data base of the PDA. After each test, the information (sampling location, operator, sample ID, etc) should be input into the data base for that sample.

9.5 Instrument and iPAQ Shutdown

1. Following the last analytical sequence, select file located at the bottom left hand corner of the iPAQ and select exit.

2. Select file and exit again until the start screen appears then turn off the iPAQ by selecting the button in the upper right corner.

3. Turn off the XRFA main power (on/off switch located on back of analyzer).

4. Remove the iPAQ from the XRFA and place both units in the water proof case for safe storage.

9.6 XRF Interference Calculation

The below procedure corrects for specific interferences in soils, or for matrixes other than soil, such as water, where analyses from the XRF has already been compared to laboratory (ICP) data. For example, if measurements for a specific element from the XRF are consistently elevated by a factor of 2, then the element rate within the XRF should be decreased by 2. Then the measurements from the XRF will match the laboratory data.

1. In soil mode, at the Standardization Screen, choose Options

2. Next, choose Element Rates

3. Enter the administrator password – lower case z

4. Change the Factor Number of the element by the percentage the XRF measurements are consistently different from the laboratory measurements. For example, if arsenic readings using the XRF are always twice the amount of the laboratory analysis, and the Factor Number for arsenic is 1500, then the new Factor Number for arsenic should be 750. This function should not be adjusted. If the values are that far off then the instrument needs to be sent back to the

manufacturer for maintenance

10.0 DATA RECORDS AND MANAGEMENT

All XRFA printouts, worksheets, logbook entries, and associated documentation must be filed in the project folder. Copies of the documentation may be required in submitted data reports. Any maintenance performed should be recorded in the XRFA Instrument Logbook.

11.0 QUALITY CONTROL

11.1 Internal Calibration Check

The energy calibration check is performed automatically during the initial standardization procedure when the instrument is started.

The software does not allow the XRFA to be used if the energy calibration check procedure fails. The energy resolution and source strength information is recorded into the XRFA log book.

11.2 Calibration Verification Check (CVC)

A calibration verification sample is used to check the accuracy of the instrument and to assess the stability and consistency of results for the analytes of interest.

A sample control check is analyzed following the initial standardization procedure of each working day, during sample analysis, and at the end of the day.

The frequency of the checks during analysis and which standards will be used for the check is DQO dependent. It is recommended to check every 20 samples or after four hours of continual use, whichever occurs first.

The measured value for each target analyte should be ± 20% of the value considered accurate in order for the CVC to pass. Refer to Appendix I for a list of analytes and their true values.

If the %D falls outside of this acceptance range, the analyzer must be restandardized followed by an acceptable CVC before analysis can continue. The batch of samples analyzed before the unacceptable CVC must be reanalyzed.

11.3 Instrument Blank

An instrument blank verifies that no contamination exists in the spectrometer or on the probe window. The instrument blank should be analyzed daily, following the standardization and CVC, every twenty samples or four hours of continual use, whichever occurs first, and at the end of the day.

All target analyte concentrations should be less than its MDL. The detection limits of the elements detected by the XRFA are given in Attachment III. If concentrations exceed these limits, then the probe window should be checked for contamination.
Clean the probe window and reanalyze the instrument blank. If contamination is not the problem,

zero the XRFA as detailed in the user manual.

Once the window is cleaned or the instrument is zeroed, the XRFA should be re-standardized and verified with acceptable CVC and instrument blank check before analyzing samples. If acceptable, re-analyze the samples prior to the last acceptable blank check.

11.4 Method Blank

The method blank is used to monitor for contaminates during preparations of samples and undergoes the same preparation procedure as the samples. The concentration of all required analytes should be less than its MDL and should be analyzed as required by site-specific DQOs.

A method blank is only required if samples are prepared according to section 9.3. If the concentration exceeds its MDL, identify the problem and all samples associated with the method blank must be reanalyzed.

11.5 Method Precision

A minimum of one sample (NIST 2709) per project should be analyzed at least seven times in replicate unless instrument drift or maintenance has occurred, with each measurement made for the same analysis time as for the field samples.

For the XRF data to be considered adequately precise, the relative standard deviation (RSD) for each analyte should not be greater than 20% except for Cr. The RSD value for Cr should not be greater than 30%. Precision requirements are defined in the site-specific SAP/QAPP.

11.6 Confirmation Samples

To verify XRF data, laboratory analysis is performed on a portion of the XRF analyzed samples as required by the site-specific SAP/QAPP.

The confirmation samples are splits of the homogenous sample material analyzed by the XRFA. Submit a minimum of 10% of the total samples for laboratory confirmation analysis.

12.0 DATA ANALYSIS AND CALCULATIONS

The XRFA instrument has software capable of storing all analytical results and spectra. The instrument software automatically performs all calculations including minimum detection levels and 95% confidence intervals.

Calculations:

Method Precision RSD = (SD/Mean Concentration) x 100

Where:
RSD = Relative standard deviation for the precision measurement of the analyte
SD = Standard deviation of the concentration for the analyte
Mean Concentration = Mean concentration for the analyte

13.0 REFERENCES

Method 6200, Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment, Revision 0, January 1998.

Innov-X Systems Alpha Series X-Ray Fluorescence Spectrometers Version 2.1, August 2005.

14.0 APPENDIX

Appendix I: NIST 2710 Reference Material (Certified)
Appendix II: NIST 2710 Reference Material (Non-Certified)
Appendix III: RTC XRF-408 Certificate of Analysis
Appendix IV: Target Analyte List

Appendix I: NIST 2710 Reference Material (certified)

Table 1. Certified Value

Element	Mass Fraction (%)	Element	Mass Fraction (mg/kg)
Aluminum	6.44 ± 0.08	Antimony	38.4 ± 3
Calcium	1.25 ± 0.03	Arsenic	626 ± 38
Iron	3.38 ± 0.10	Barium	707 ± 51
Magnesium	0.853 ± 0.042	Cadmium	21.8 ± 0.2
Manganese	1.01 ± 0.04	Copper	2950 ± 130
Phosphorus	0.106 ± 0.015	Lead	5532 ± 80
Potassium	2.11 ± 0.11	Mercury	32.6 ± 1.8
Silicon	28.97 ± 0.18	Nickel	14.3 ± 1.0
Sodium	1.14 ± 0.06	Silver	35.3 ± 1.5
Sulfur	0.240 ± 0.006	Vanadium	76.6 ± 2.3
Titanium	0.283 ± 0.010	Zinc	6952 ± 91

Noncertified Values: Noncertified values shown below are provided for information only. An element concentration value is not certified if a bias is suspected in one or more of the methods used for certification, or if two independent methods are not available.

Appendix II: NIST 2710 Reference Material (noncertified)

Table 2. Noncertified Values

Element	Mass Fraction (%)	Element	Mass Fraction (mg/kg)
Carbon	3	Bromine	6
		Cerium	57
		Cesium	107
		Chromium	39
		Cobalt	10
		Dysprosium	5.4
		Europium	1
		Gallium	34
		Gold	0.6
		Hafnium	3.2
		Holmium	0.6
		Indium	5.1
		Lanthanum	34
		Molybdenum	19
		Neodymium	23
		Rubidium	120
		Samarium	7.8
		Scandium	8.7
		Strontium	330
		Thallium	1.3
		Thorium	13
		Tungsten	93
		Uranium	25
		Ytterbium	1.3
		Yttrium	23

Appendix III: RTC XRF-408 Certificate of Analysis

Analyte: **Metals in Soil**	Solvent: **Soil**
Product Number: **XRF-408**	Hazard: **Irritant**
	Lot: **010291**

	Certified Value	Verification Value	Units	Uncertainty	Verified to NIST SRM
Arsenic, As	500	501	mg/Kg	15.2	3103a
Barium, Ba	500	500	mg/Kg	15.2	3104a
Cadmuim, Cd	500	500	mg/Kg	15.1	3108
Chromium, Cr	500	500	mg/Kg	15.1	3112a
Lead, Pb	500	500	mg/Kg	15.1	3128
Mercury, Hg	500	501	mg/Kg	15.1	3133
Selenium, Se	500	500	mg/Kg	15.1	3149
Silver, Ag	500	500	mg/Kg	15.1	3151

The certified concentration is based upon gravimetric procedures. The uncertainty associated with the certified value is +/- 0.3% relative, which is the sum of the estimated errors due to the purity of the raw material, the gravimetric preparation of the solution, and losses due to packaging.

Traceability: The standard was manufactured under an ISO 9001 registered quality system. The balance used to weigh raw materials is accurate to +/- 0.0001g and calibrated regularly using mass standards, which are traceable to NST. All dilutions are performed gravimetrically. The standard is directly traceable to NIST SRM's as listed above.

Appendix IV: Target Analyte List

OLYMPUS®

Your Vision, Our Future

HANDHELD XRF ANALYZERS

Limits of Detection

Low-Density Sample Types - (soils, powders, liquids)

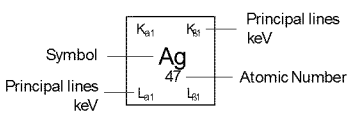
Not Measured | <1 % | <0.5 % | <200 ppm | <50 ppm | <20 ppm | <10 ppm | <5 ppm

	IIA	IIIB	IVB	VB	VIB	VIIB	Group VIII			IB	IIB	IIIA	IVA	VA	VIA	VIIA	
H 1																	He 2
0.05 Li 3	0.11 Be 4											0.18 B 5	0.28 C 6	0.39 N 7	0.52 O 8	0.68 F 9	0.85 Ne 10
1.04 1.07 Na 11	1.25 1.3 Mg 12											1.49 1.56 Al 13	1.74 1.84 Si 14	2.01 2.14 P 15	2.31 2.46 S 16	2.62 2.82 Cl 17	2.96 3.19 Ar 18
3.31 3.59 K 19	3.69 4.01 Ca 20 0.34 0.34	4.09 4.46 Sc 21 0.4 0.4	4.51 4.93 Ti 22 0.45 0.46	4.95 5.43 V 23 0.51 0.52	5.41 5.95 Cr 24 0.57 0.58	5.9 6.49 Mn 25 0.64 0.65	6.4 7.06 Fe 26 0.71 0.72	6.93 7.65 Co 27 0.78 0.79	7.48 8.26 Ni 28 0.85 0.87	8.05 8.91 Cu 29 0.93 0.95	8.64 9.57 Zn 30 1.01 1.03	9.25 10.26 Ga 31 1.1 1.12	9.89 10.98 Ge 32 1.19 1.22	10.54 11.73 As 33 1.28 1.32	11.22 12.5 Se 34 1.38 1.42	11.92 13.29 Br 35 1.48 1.53	12.65 14.11 Kr 36 1.59 1.64
13.4 14.96 Rb 37 1.69 1.75	14.17 15.84 Sr 38 1.81 1.87	14.96 16.74 Y 39 1.92 2	15.78 17.67 Zr 40 2.04 2.12	16.62 18.62 Nb 41 2.17 2.26	17.48 19.61 Mo 42 2.29 2.39	18.37 20.62 Tc 43 2.42 2.54	19.28 21.66 Ru 44 2.56 2.68	20.22 22.72 Rh 45 2.83	21.18 23.82 Pd 46 2.84 2.99	22.16 24.94 Ag 47 2.98 3.15	23.17 26.1 Cd 48 3.13 3.32	24.21 27.28 In 49 3.29 3.49	25.27 28.49 Sn 50 3.44 3.66	26.36 29.73 Sb 51 3.6 3.84	27.47 31 Te 52 3.77 4.03	28.61 32.29 I 53 3.94 4.22	29.78 33.62 Xe 54 4.11 4.42
30.97 34.99 Cs 55 4.29 4.62	32.19 36.38 Ba 56 4.47 4.83		55.79 63.23 Hf 72 7.9 9.02	57.53 65.22 Ta 73 8.15 9.34	59.32 67.24 W 74 8.4 9.67	61.14 69.31 Re 75 8.65 10.01	63 71.41 Os 76 8.91 10.36	64.9 73.56 Ir 77 9.18 10.71	66.83 75.75 Pt 78 9.44 11.07	68.8 77.98 Au 79 9.71 11.44	70.82 80.25 Hg 80 9.99 11.82	72.87 82.58 Tl 81 10.27 12.21	74.97 84.94 Pb 82 10.55 12.61	77.11 87.34 Bi 83 10.84 13.02	79.29 89.8 Po 84 11.13 13.45	81.52 92.3 At 85 11.43 13.88	83.78 94.87 Rn 86 11.73 14.32
86.1 97.47 Fr 87 12.03 14.77	88.47 100.13 Ra 88 12.34 15.24																

Lanthanides 57-71	33.44 37.8 La 57 4.65 5.04	34.72 39.26 Ce 58 4.84 5.26	36.03 40.75 Pr 59 5.03 5.49	37.36 42.27 Nd 60 5.23 5.72	38.72 43.83 Pm 61 5.43 5.96	40.12 45.41 Sm 62 5.64 6.21	41.54 47.04 Eu 63 5.85 6.46	43 48.7 Gd 64 6.06 6.71	44.48 50.38 Tb 65 6.27 6.98	46 52.12 Dy 66 6.5 7.25	47.55 53.88 Ho 67 6.72 7.53	49.13 55.68 Er 68 6.95 7.81	50.74 57.52 Tm 69 7.18 8.1	52.39 59.37 Yb 70 7.42 8.4	54.07 61.28 Lu 71 7.66 8.71
Actinides 89-103	90.88 102.85 Ac 89 12.65 15.71	93.35 105.61 Th 90 12.97 16.2	95.87 108.43 Pa 91 13.29 16.7	98.44 111.3 U 92 13.61 17.22	101.00 114.18 Np 93 13.95 17.74	103.65 117.15 Pu 94 14.28 18.28	106.35 120.16 Am 95 14.62 18.83	109.10 123.24 Cm 96 14.96 19.39	111.90 126.36 Bk 97 15.31 19.97	114.75 129.54 Cf 98 15.66 20.56	117.65 132.78 Es 99 16.02 21.17	120.60 136.08 Fm 100 16.38 21.79	Md 101	No 102	Lr 103

Alloy Analysis:
Elements detected: Magnesium (Mg, Z=12) through Sulfur (S, Z=16) and Titanium (Ti, Z=22) through Plutonium (Pu, Z=94).

Please see separate Alloy Analysis LOD Speci cations.



INNOV-X

Detection limits are a function of testing time, sample matrix and presence of interfering elements.
Detection limits are estimates based on 1-2 minutes test times and detection con dence of 3σ (99.7% con dence).
Interference-free detection limits are intended as guidelines: please contact Olympus Innov-X to discuss your speci c application.

PHOTON ENERGIES, IN ELECTRON VOLTS, OF PRINCIPAL K- AND L-SHELL EMISSION LINES

Element	Symbol	Atomic #	$K_{\beta 1}$	$K_{\gamma 1}$	$L_{\beta 1}$	$L_{\gamma 1}$
Actinium	Ac	89	90.88	102.85	12.65	15.71
Aluminum	Al	13	1.49	1.56	0	0
Antimony	Sb	51	26.36	29.73	3.6	3.84
Argon	Ar	18	2.96	3.19	0	0
Arsenic	As	33	10.54	11.73	1.28	1.32
Astatine	At	85	81.52	92.3	11.43	13.88
Barium	Ba	56	32.19	36.38	4.47	4.83
Beryllium	Be	4	0.11	0	0	0
Bismuth	Bi	83	77.11	87.34	10.84	13.02
Boron	B	5	0.18	0	0	0
Bromine	Br	35	11.92	13.29	1.48	1.53
Cadmium	Cd	48	23.17	26.1	3.13	3.32
Calcium	Ca	20	3.69	4.01	0.34	0.34
Carbon	C	6	0.28	0	0	0
Cerium	Ce	58	34.72	39.26	4.84	5.26
Cesium	Cs	55	30.97	34.99	4.29	4.62
Chlorine	Cl	17	2.62	2.82	0	0
Chromium	Cr	24	5.41	5.95	0.57	0.58
Cobalt	Co	27	6.93	7.65	0.78	0.79
Copper	Cu	29	8.05	8.91	0.93	0.95
Dysprosium	Dy	66	46	52.12	6.5	7.25
Erbium	Er	68	49.13	55.68	6.95	7.81
Europium	Eu	63	41.54	47.04	5.85	6.46
Fluorine	F	9	0.68	0	0	0
Francium	Fr	87	86.1	97.47	12.03	14.77
Gadolinium	Gd	64	43	48.7	6.06	6.71
Gallium	Ga	31	9.25	10.26	1.1	1.12
Germanium	Ge	32	9.89	10.98	1.19	1.22
Gold	Au	79	68.8	77.98	9.71	11.44
Hafnium	Hf	72	55.79	63.23	7.9	9.02
Holmium	Ho	67	47.55	53.88	6.72	7.53
Indium	In	49	24.21	27.28	3.29	3.49
Iodine	I	53	28.61	32.29	3.94	4.22
Iridium	Ir	77	64.9	73.56	9.18	10.71
Iron	Fe	26	6.4	7.06	0.71	0.72
Krypton	Kr	36	12.65	14.11	1.59	1.64
Lanthanum	La	57	33.44	37.8	4.65	5.04
Lead	Pb	82	74.97	84.94	10.55	12.61
Lithium	Li	3	0.05	0	0	0
Lutetium	Lu	71	54.07	61.28	7.66	8.71
Magnesium	Mg	12	1.25	1.3	0	0
Manganese	Mn	25	5.9	6.49	0.64	0.65
Mercury	Hg	80	70.82	80.25	9.99	11.82
Molybdenum	Mo	42	17.48	19.61	2.29	2.39
Neodymium	Nd	60	37.36	42.27	5.23	5.72

Element	Symbol	Atomic #	$K_{\beta 1}$	$K_{\gamma 1}$	$L_{\beta 1}$	$L_{\gamma 1}$
Neon	Ne	10	0.85	0	0	0
Nickel	Ni	28	7.48	8.26	0.85	0.87
Niobium	Nb	41	16.62	18.62	2.17	2.26
Nitrogen	N	7	0.39	0	0	0
Osmium	Os	76	63	71.41	8.91	10.36
Oxygen	O	8	0.52	0	0	0
Palladium	Pd	46	21.18	23.82	2.84	2.99
Phosphorus	P	15	2.01	2.14	0	0
Platinum	Pt	78	66.83	75.75	9.44	11.07
Polonium	Po	84	79.29	89.8	11.13	13.45
Potassium	K	19	3.31	3.59	0	0
Praseodymium	Pr	59	36.03	40.75	5.03	5.49
Promethium	Pm	61	38.72	43.83	5.43	5.96
Protactinium	Pa	91	95.87	108.43	13.29	16.7
Radium	Ra	88	88.47	100.13	12.34	15.24
Radon	Rn	86	83.78	94.87	11.73	14.32
Rhenium	Re	75	61.14	69.31	8.65	10.01
Rhodium	Rh	45	20.22	22.72	2.7	2.83
Rubidium	Rb	37	13.4	14.96	1.69	1.75
Ruthenium	Ru	44	19.28	21.66	2.56	2.68
Samarium	Sm	62	40.12	45.41	5.64	6.21
Scandium	Sc	21	4.09	4.46	0.4	0.4
Selenium	Se	34	11.22	12.5	1.38	1.42
Silicon	Si	14	1.74	1.84	0	0
Silver	Ag	47	22.16	24.94	2.98	3.15
Sodium	Na	11	1.04	1.07	0	0
Strontium	Sr	38	14.17	15.84	1.81	1.87
Sulfur	S	16	2.31	2.46	0	0
Tantalum	Ta	73	57.53	65.22	8.15	9.34
Technetium	Tc	43	18.37	20.62	2.42	2.54
Tellurium	Te	52	27.47	31	3.77	4.03
Terbium	Tb	65	44.48	50.38	6.27	6.98
Thallium	Tl	81	72.87	82.58	10.27	12.21
Thorium	Th	90	93.35	105.61	12.97	16.2
Thulium	Tm	69	50.74	57.52	7.18	8.1
Tin	Sn	50	25.27	28.49	3.44	3.66
Titanium	Ti	22	4.51	4.93	0.45	0.46
Tungsten	W	74	59.32	67.24	8.4	9.67
Uranium	U	92	98.44	111.3	13.61	17.22
Vanadium	V	23	4.95	5.43	0.51	0.52
Xenon	Xe	54	29.78	33.62	4.11	4.42
Ytterbium	Yb	70	52.39	59.37	7.42	8.4
Yttrium	Y	39	14.96	16.74	1.92	2
Zinc	Zn	30	8.64	9.57	1.01	1.03
Zirconium	Zr	40	15.78	17.67	2.04	2.12

COLLECTION, ANALYSIS AND DISPOSAL OF ESAT LABORATORY WASTE

APPROVAL PAGE

Written By: David B Roberts Jr — 1-18-07
ESAT Senior Analytical Chemist — Date

ESAT Review: 1/17/2007
ESAT QA/QC Coordinator — Date

ESAT Approval: 1/17/2007
ESAT Regional Manager — Date

EPA Review:
Gregory Saunders — Date
EPA Region 8 Laboratory Health and Safety

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1.0 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure describes the procedures and practices for safely collecting, analyzing and storing aqueous corrosive wastes.

1.2 This procedure is applicable to the following aqueous waste streams; acidic instrument waste, acidic reagents (except standards), sample digestates and samples preserved for analysis with acid.

1.3 This procedure is applicable to solid wastes such as soils, vegetation and biota samples.

2.0 SUMMARY OF PROCEDURE

2.1 Aqueous sample, digestates, reagents and some instrument waste contain small amounts of mineral acids.

2.1.1 The presence of these acids causes the pH of the waste to be below 2 and hence be defined as hazardous.

2.1.2 In addition, these wastes may contain metal concentrations which exceed discharge standards.

2.2 Solid wastes may contain metal concentrations which exceed disposal standards.

2.2 This waste must be properly labeled, contained and stored in accordance with all state, federal and ESAT regulations.

2.2.1 This procedure includes the initiation of satellite waste containers, documentation accompanying the waste and procedures for placing the waste in the designated waste storage area.

3.0 DEFINITIONS / ACRONYMS

3.1 Aqueous Corrosive Waste - An aqueous solution with a pH<2.

3.2 Evidentiary Materials – Samples, sample containers and sample residuals.

3.3 F Waste: Hazardous waste from nonspecific sources (See 40 CFR 261.31.).

3.4 Satellite Waste Container - A container used to collect waste during generation.

3.5 Secondary Containment

3.5.1 A second level of containment ensuring no release if the initial containment fails.

3.6 Waste Control Officer (WCO)

3.6.1 Person responsible for maintaining control of all documentation of waste disposal.

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4.0 HEALTH AND SAFETY

4.1 All pertinent procedures outlined in the EPA Region 8 Chemical Hygiene Plan (CHP) will be followed in performance of the handling of wastes.

4.1.1 The use of laboratory equipment and chemicals exposes the analyst to several potential hazards.

4.1.2 Good laboratory technique and safety practices should be followed at all times.

4.2 Solutions classified as aqueous corrosive wastes normally contain percentage levels of mineral acids and can contain certain inorganic elements known to be hazardous.

4.2.1 Gloves, protective eye wear and laboratory coats should be worn at all times when handling samples, reagents, or when in the vicinity of others handling these items.

4.3 Satellite waste containers must always be tightly capped when not in use.

4.4 Satellite waste containers can weigh in excess of 50 lbs and should be lifted carefully.

4.5 Spilled samples, reagents, and water should be cleaned up from instrument and autosampler surfaces immediately.

4.5.1 In the case of acid spills, the acid should be neutralized with acid spill kits available in the laboratory.

5.0 CAUTIONS

5.1 Prior to starting work that involves handling wastes, personnel should review the project plan safety requirements, analytical procedure safety requirements, and this waste management procedure.

5.1.1 When in doubt as to the proper procedure to follow, contact the Health and Safety Officer for guidance.

5.1.2 Personnel should minimize exposure to potential health hazards through the use of engineering and administrative controls, work practice procedures and practices, and proper protective equipment.

6.0 PERSONNEL QUALIFICATIONS

6.1 Personnel Responsibilities

6.1.1 Personnel responsibilities for hazardous waste management at EPA Region 8 laboratory are described in the Introduction/Executive Summary of the *Safety, Health and Environmental Management Program (SHEMP) Manual* (Section 2.2 Hazardous Waste Management).

6.2 Personnel Training

6.2.1 Federal and state regulations require that employees who handle hazardous waste be provided with initial and annual training. Initial orientation and on-the-job training are provided to new ESAT employees within their first month of employment, and refresher training is provided on an annual basis thereafter.

6.2.2 This training is designed to keep employees familiar with waste handling procedures in place at EPA, along with applicable regulations.

6.2.3 Training completion will be enforced by the supervisor and documented for each

individual by the ESAT Health and Safety Officer.

7.0 EQUIPMENT AND SUPPLIES

7.1 The WCO (Waste Control Officer) will assure that a supply of appropriate waste containers and labels are available for use.

7.2 Waste containers must be able to be tightly capped and both the container and the secondary containment must be chemically resistant to corrosive materials.

8.0 SATELLITE WASTE CONTAINER PREPARATION

8.1 The waste container will be properly labeled as appropriate.

- 8.1.1 A Waste Container ID is assigned to the container by the person who initiates the container's designation.
  - 8.1.1.1 The container ID will be written on the container with a Sharpie in such a location as to be easily seen.
- 8.1.2 The Container ID will follow the YYMMDD-XXXX-# format where "XXXX" describes the laboratory room number and the "#" will be the number of the container generated on that particular day.
- 8.1.3 Affix an appropriate red and white hazardous label for containers used for suspected or known hazardous materials.
- 8.1.4 Affix a black and white label warning of the presence of corrosives (pH<2) when the contents to be added to the container are known to be acidic.

8.2 A waste container inventory log will be initiated and placed next to the container.

- 8.2.1 An attached (or located in the near vicinity) hazardous waste container inventory log, listing the accumulated waste maintained by the generator(s).
- 8.2.2 The waste inventory sheet should include the container ID number and its date, description and amount of waste added, date of the addition, and the name of the person making each addition.

Note: The waste inventory sheet serves two important purposes. It guards against addition of incompatible chemicals to the container mix and allows packers to determine the correct classification of the waste for transport and disposal.

9.0 WASTE COLLECTION AND ANALYSIS

9.1 Waste must be collected as near as possible to the point of generation and have secondary containment.

9.2 Containers must be kept closed except when waste is being added.

9.3 When a container of waste is approximately 85% full, the waste inventory sheet must be signed, dated and entered into the inventory system by the WCO.

9.4 Full aqueous waste containers will be sub-sampled, the waste inventory sheet filled out and transported to the waste storage area within 24 hours.

- 9.4.1 The waste inventory sheet must be clearly labeled as "Awaiting analysis".

9.5 Waste containers that are full or otherwise ready for disposal will be transported to the F wing where the wastes are segregated by waste categories: D00I, ignitable liquids; D002, corrosive materials; D003, reactive materials; and D004 to D043, toxic materials.

9.5.1 Each unit will be labeled with the start of accumulation date and, as appropriate, a "hazardous waste" or "non-hazardous waste" label.

9.6 Analysis of each aqueous waste container must be completed within 10 days.

9.6.1 The results are then entered on the waste inventory sheet.

9.7 Solid wastes designated for disposal are collected in an approved container.

9.7.1 In lieu of analyzing the solids, the data collected during the analysis of the samples is used to indicate the level, if any, of metals concentration in the solid waste.

9.7.2 In general, segregating the solids into LIMS workorder specific groups will make calculating the metals concentration less complicated.

9.8 A copy of the completed waste inventory sheet along with the raw data is provided to the WCO and the original is attached to the waste container.

10.0 DATA AND RECORDS MANAGEMENT

10.1 At EPA Region 8 laboratory, waste is tracked through the use of container labels, waste container logs, an in-house tracking system, physical inventories, hazardous waste shipping manifests, and certificates of disposal.

10.2 Data from waste container sheets for wastes generated in the laboratory is entered into the Hazardous Waste Tracking System (HWTS) bound green notebook by the WCO, at the time of transport to the F wing.

11.0 WASTE MINIMIZATION

11.1 Metals laden waste volumes are minimized by the use of a dedicated waste receptacle in which no other laboratory waste is placed.

11.1.1 Waste concentrations are minimized by judicious use of metals standard solutions and materials.

11.1.2 In addition, ESAT chemists are working with field personnel to reduce the amount of excess sample collected.

12.0 REFERENCES

12.1 EPA Region 8, Chemical Hygiene Plan, current version.

12.2 EPA Region 8, Health and Safety Plan, current version.

12.3 EPA Region 8, Waste Management SOP, current version.

SAMPLE RECEIPT, CUSTODY, STORAGE AND LIMS ENTRY OF SAMPLES

APPROVAL PAGE

Written By: _____ 05/06/2009
ESAT Senior Analytical Chemist — Date

ESAT Review: _____ 5/6/2009
ESAT QA/QC Coordinator — Date

ESAT Approval: _____ 5/6/09
ESAT Region 8 Manager — Date

This document has been prepared by the TechLaw Inc. ESAT Region 8 Team and is intended to provide documentation of administrative, analytical and quality control procedures used in the daily performance of ESAT support services. This is a controlled document and may not be provided to a third party, such as consultants or other governmental agencies, unless said third party agrees that the recipient agrees to keep confidential the contents of this document. If the document is provided as a controlled document, the user agrees to surrender the document upon request of TechLaw, Inc. If the document is provided as an uncontrolled document, the user understands that subsequent revisions will not be provided.

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1.0 SCOPE AND APPLICABILITY

1.1 The purpose of this standard operating procedure (SOP) is to establish a safe, traceable, and consistent laboratory process for receiving, tracking, and storage of ESAT samples at the USEPA Region 8 Laboratory. These may include surface waters, ground waters, soils, sediments and biological materials.

2.0 SUMMARY OF PROCEDURE

2.1 This SOP specifies the requirements for project definition, sample receipt, control, and record keeping by ESAT in the USEPA Region 8 Laboratory. The following objectives are defined in detail within this document:

2.2 EPA Project Definition - Prior to accepting samples at the laboratory, an agreement between the EPA Project Officer (PO) and the laboratory must be set forth in a Technical Direction Form (TDF) which details the required analytical methods, target analytes, approximate quantity of samples, receipt date, analytical quality control procedures and data deliverables.

2.3 ESAT Project Definition - Following receipt of the TDF, ESAT personnel will create a project in LIMS that defines the requirements detailed in the TDF.

2.4 Sample Integrity Inspection
- 2.4.1 The samples listed on the chain of custody (COC) are compared to the received samples to ensure all samples listed on the COC were received by the laboratory.
- 2.4.2 The samples and shipping coolers are inspected for leakage or breakage.
- 2.4.3 The temperature of the samples upon receipt is recorded.
- 2.4.4 The sample preservation is verified.
- 2.4.5 Any breach of the sample integrity will be noted and become a part of the project record.

2.5 Chain of Custody (COC) Verification
- 2.5.1 The COC establishes a traceable, legal record of the possession of the samples from sampling through analysis.
- 2.5.2 Laboratory personnel will compare the sample identification as listed on the COC to the identification on the samples.
- 2.5.3 Identify any sample requiring analyses that have short holding times and notify laboratory personnel of the sample arrival.
- 2.5.4 All discrepancies will be noted and become part of the project record.
- 2.5.5 Sample custody is maintained by storing the samples in a locked cooler.
- 2.5.6 Movement of the samples in and out of the cooler is tracked in a logbook.

2.6 ESAT LIMS Sample Log-In - After completing the sample receipt procedures, the samples are logged into the ESAT LIMS by utilizing the ESAT project definition and either an electronic XML file or by hand entering sample information from the COC and any noted discrepancies.

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3.0 ACRONYMS AND DEFINITIONS

°C	Degrees Celsius
COC	Chain of Custody
DM	Dissolved Metals
ESAT	Environmental Services Assistance Team
ID	Identification
LIMS	Laboratory information (data) management system
PO	Project Officer
PPE	Personal protective equipment
QA	Quality Assurance
QC	Quality Control
SOP	Standard operating procedure
TDF	Technical Directive Form
TR	Total Recoverable (Metals Fraction)
USEPA	United States Environmental Protection Agency

4.0 HEALTH AND SAFETY

4.1 The receiving personnel shall ensure the sample login area is clean and free of any potential contaminants prior to working in the area.

4.2 Proper personal protective equipment (PPE) is required for receipt of samples. This includes gloves, eye protection, and a lab coat at the minimum.

4.3 Leaking containers can pose a health risk due to the possible presence of acids, toxic components, etc., making inhalation of toxic vapors a possible hazard.
- 4.3.1 All coolers should be opened in a room with adequate ventilation.
- 4.3.2 If broken sample containers are present, additional PPE and engineering controls, e.g. fume hood, may be required. If the use of spill cleanup material is necessary, the proper method of cleanup and disposal must be followed.
- 4.3.3 Assistance from the ESAT or EPA Health & Safety Officer for proper handling and disposal procedures may be required.

4.4 Sample receipt personnel should be familiar with the location of additional safety equipment.
- 4.4.1 Spill and neutralizer equipment are available in the sample receipt area.
- 4.4.2 Eye wash and safety shower in the sample receiving area should be verified as unobstructed prior to unpacking the samples.

5.0 PERSONNEL QUALIFICATIONS

5.1 ESAT Team Members
- 5.1.1 The receiving and checking of incoming samples must be performed by an Environmental Services Assistance Team (ESAT) member trained in the proper performance of this SOP.
- 5.1.2 The sample receiver must be familiar with interpreting COC documentation, performing pH determinations, and maintaining custody of samples.

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5.1.3 Personnel receiving samples should have a baseline physical examination performed prior to receiving samples.
5.1.4 Some lifting of 30-50 pound coolers/containers may be required.

5.2 EPA Personnel
5.2.1 Periodically, EPA personnel will move coolers containing samples for ESAT into the ESAT sample storage cooler.
5.2.2 EPA personnel will notify ESAT team members of the arrival of the samples.

6.0 APPARATUS AND MATERIALS

6.1 Calibrated and Certified Thermometers
6.1.1 Thermometers used for measuring sample storage cooler temperatures are calibrated annually under the supervision of the EPA laboratory quality assurance officer using a certified thermometer.
6.1.2 An infrared temperature indicator is maintained and used by ESAT personnel for recording the temperature of the samples upon arrival.

6.2 Wide-range pH Paper.

6.3 Waste container, properly labeled.

6.4 Promium ELEMENT Laboratory Information Management System (LIMS) for sample tracking and reporting.

6.5 Laboratory fume hood for opening sample coolers.

6.6 Refrigerated and secured sample storage cooler.

7.0 COOLER RECEIPT AND ACCEPTANCE

7.1 Sample Integrity Inspection
7.1.1 Generally, samples are received through the main entrance of the facility via Fed Ex or delivery from the sampling contractor.
7.1.1.1 Save the copy of the air bill associated with each cooler and place it in the project folder.
7.1.1.2 The method of delivery is noted in the “Remarks” section of the COC and later is indicated in the project when the samples are logged into the LIMS.
7.1.2 Retrieve a sample cart from the metals instrument room (B-104) and move the coolers to the sample receipt area in the facility.
7.1.3 Examine the shipping coolers for any damage or leaks and note their presence for inclusion into the project folder.
7.1.4 Open the cooler(s) while the cooler is located under the exhaust hood in the sample receipt area.
7.1.5 Remove the COC from the cooler.
7.1.6 Sign the “Received” section of each page of the COC and indicate the data and time of the receipt.

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Note: Ideally, the sampler or customer should be present during the transition of the samples into ESAT custody including opening of the coolers and cross-checking of information, whenever possible.

7.1.7 Unpack the cooler using the COC to organize the samples on the work table in the sample receipt area.

7.1.7.1 If any issues with sample integrity are observed (e.g., damage to the sample container, contamination, etc.), the analyst should discuss this in the case narrative so that data users are aware that the sample may have been compromised.

7.1.7.2 Any correspondence with and direction received from the EPA PO regarding a compromised sample should be received in writing via email, and that email should be included in the data package.

7.1.8 Temporarily place the ice or baggies filled with ice in the deep sink under the exhaust hood.

7.1.9 Using the IR thermometer, measure the temperature of the first unpacked sample. This temperature will be recorded into LIMS and original COC.

7.1.10 Inspect each sample container for damage or leaking and note any circumstance for inclusion in the project folder.

7.1.11 Verify the preservation of any samples that are indicated on the COC as having been preserved to a specific pH.

7.1.12 Hold a piece of wide range pH paper over the waste container and pour 3-5 mL of each sample over the pH paper and into the waste container.

7.1.13 If the sample is properly preserved, no further action is required.

7.1.14 Recap the sample container and proceed with the log-in procedure.

Note: The sample aliquots used to check the pH must be disposed of in the metals waste container in room B-104.

7.1.15 Improperly preserved samples will be properly preserved before placing into the storage cooler. Carefully note on the COC and case narrative which samples were not properly preserved.

7.2 Chain of Custody (COC) Verification

7.2.1 The samples should be accompanied by a COC, sample tags, and custody seals.

7.2.1.1 All information required on the forms and tags must be properly completed and legible.

7.2.1.2 The sample tag information must be verified against the corresponding sample information noted on the COC.

7.2.2 In the case of COC discrepancies, the sample ID Tag will be assumed as the true information and the discrepancies must be clearly noted on the COC with the login personnel's initials and date.

7.2.2.1 All COC discrepancies should be discussed in the case narrative of the data package.

7.2.2.2 If a COC discrepancy requires contact with the EPA PO, this should also be discussed in the case narrative of the data package. If COC discrepancies are resolved verbally with the EPA PO, an email should be sent to confirm the reconciliation of discrepancies and a copy of the email should be included in the data package with the COC.

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7.2.3 If the documentation is incomplete, the ESAT Manager and EPA PO will be contacted regarding the discrepancy. The EPA PO will decide if the process will continue.
7.2.4 After each sample is unpacked from the shipping container and the sampling information is verified, it is segregated into various storage trays by analysis.
7.2.5 The trays are labeled with a tag in a plastic shield with the project name, LIMS number, TDF number, due date, and requested analysis.
7.2.6 The labeled trays are then placed in the walk-in cooler "A" and secured by locking the cooler with the provided padlock.
7.2.7 The trays are removed by the analyst prior to analysis.
7.2.8 The analysts record the removal of the samples from the cooler by using the logbook in the sample receipt area.
7.2.9 Empty the plastic bags filled with ice that were placed in the sink and put the empty bags into the provided waste container in the sample receipt area.

8.0 PROJECT CREATION AND SAMPLE ENTRY IN LIMS

8.1 ESAT Project Creation in LIMS
8.1.1 Open the LIMS software.
8.1.2 In the Project Management dropdown menu, select "Projects".
8.1.3 Highlight a similar project and be sure to check it has the required test codes.
8.1.4 Select the "Copy" option.
8.1.5 Double click the "Superfund" client option.
8.1.6 Rename the project in the dialog window.
8.1.7 From the new project screen select "Edit".
8.1.8 Put the TDF number in both the "Project Number" and "PO number" fields.
8.1.9 Select the "project Manager" from the drop down menu.
8.1.10 Check that the default EDD is "StdESATExel_rev1.exe".
8.1.11 Enter the appropriate project name in the comments field.
8.1.12 If the test codes for the new project need to be changed, double click on "Test Codes" and from the drop down menu select the correct test codes for this project.
8.1.13 Save the project.

8.2 Work Order Creation in LIMS
8.2.1 From the "Sample Control" menu select "Work order".
8.2.2 Select "Import" and select the file location of the XML/Scribe file from the drop down menu.
8.2.3 Click the "Import" button.
8.2.4 From the "Analysis" tab match the appropriate test codes.
8.2.5 From the "Matrices" tab match the sample preservatives.
8.2.6 From the "Container" tab select "Default".
8.2.7 Click "Done" and the new work order screen will appear.

8.3 Work Order Information Editing
8.3.1 Select the Work order from the dropdown menu and click "Edit".
8.3.2 Select the project from the drop down menu in the top right corner.
8.3.3 The Project number and the PO number should match the TDF for the project.
8.3.4 In the "Submitted By" window select the appropriate sampler from the drop down menu.

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8.3.5 In the "SDG Identifier" window, type in the TDF number.
8.3.6 In the "Shipped By" window select either "Walk in" or" Fed Ex" from the drop down menu.
8.3.6.1 If shipping was by Fed Ex, enter the tracking number in that window.
8.3.7 Select the turn around time to calculate the appropriate due date for the project.
8.3.8 Check the appropriate "Condition" boxes for the samples received.
8.3.9 Ensure the Analysis Test Codes are accurate and add/delete as needed.
8.3.10 Save the Work order.

8.4 Editing Samples in the Work order
8.4.1 Click on the "Samples" tab and "Edit".
8.4.2 Verify that the sample name, container, location and comment (EPA Tag #) are correct.
8.4.3 In the "Report Matrix" drop down window select the one listed on the COC.
8.4.4 In the "Sample Type" drop down window select "Field Sample".
8.4.5 In the "Sampled By" drop down window select the one listed on the COC.
8.4.6 In the "Work Analysis" windows the test codes may or may not be applied. If more tests are needed, click the "Work Analysis" tab to see all of the available test codes.
8.4.7 Repeat sample entry/editing for all samples in the project.
8.4.8 Save the work order and click the printer icon to print the sample information.

8.5 Project Folder Creation
8.5.1 Master Project Folder
8.5.1.1 Label a new folder with the Work Order number, Project Name, TDF Number and Due Date.
8.5.1.2 Place the original COC, TDF and the shipping label in this folder.
8.5.1.3 Place the LIMS printout of the samples entered in this folder.
8.5.1.4 Place any E-mail or other documents pertaining to the project in this folder.
8.5.1.5 All analytical data will be placed in this folder until final report generation.

8.5.2 Analytical Folder
8.5.2.1 On the LIMS PC go to "Explore".
8.5.2.2 Go to the "X" drive and click on "Metals_Data_Files".
8.5.2.3 Select the appropriate year.
8.5.2.4 Go to "File", "New", "Folder".
8.5.2.5 Name the new folder using "Work Order_TDF Project Name" format. (Ex: (C606006_SC010 CalGulch June Monthly)
8.5.2.6 Repeat the file creation sequence in "WetChem_Data_Files" if the project requires this type of analysis.

8.5.3 Reporting Folder
8.5.3.1 On the ESAT PC, click and open "Explore".
8.5.3.2 Go to the "G" drive and click on "ESAT".
8.5.3.3 Click on "Analytical Reports" and then "Final Reports".
8.5.3.4 Go to "File", "New", "Folder".
8.5.3.5 Name the folder using the same convention as the Analytical Folder.

9.0 DATA AND RECORDS MANAGEMENT

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9.1 The sample checkout logbook is maintained by the EPA quality assurance/quality control QA /QC personnel.

9.2 Full logbooks will be archived, and new ones provided when necessary.

9.3 The EPA QA/QC personnel also verify thermometer calibration and log the Cooler temperatures daily.

9.4 COC records, LIMS reports, and all other correspondence become part of the ESAT retained records data file.

9.5 All entries to the sample checkout logbooks and custody records will be recorded in blue or black indelible ink.

9.6 When an entry error occurs, the author will draw a single line through the error, initial and date it, and complete the correct entry. If the space is too small for further legible entries, either the next line will be used, or the correction must be footnoted to ensure legibility of the correct entry.

9.7 Annual audits will be conducted by the ESAT QA/QC Coordinator to verify the procedures outlined in this SOP are being performed.

9.8 Refrigerated cooler temperatures are checked and recorded daily according to EPA Region 8 SOP Monitoring Refrigerator and Cooler Temperatures, current version.

10.0 WASTE MINIMIZATION

10.1 The analyzed samples are separated for consolidation and disposal. Consult the Aqueous Corrosive Waste Disposal SOP for further details (LAB-01.00_ESAT Waste Disposal_R2).

10.2 Plastic sample tag holders are reused, as are the washable trays, coolers, and carts. Sample containers are too easily contaminated and are not reused.

10.3 In order to minimize contamination of large volumes of liquids, compatible samples marked for disposal will be consolidated without further dilution.

10.4 Field coolers and some packing materials are cleaned, dried, and reused.

11.0 REFERENCES

11.1 USEPA Region 8 Laboratory SOP, Sample Receipt and Custody, current version.

11.2 ESAT Region 8 SOP, Disposal of Aqueous Corrosive Wastes.

11.3 ESAT Region 8 Health and Safety Plan, Current Version.

11.4 USEPA Region 8 Laboratory Chemical Hygiene Plan, Current Version.

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FIELD PROCEDURES — ANALYTICAL SUPPORT AND LABORATORY SELECTION

SOP Number: 02-06-05
Effective Date: 04/27/12

Technical Approval: _____Date:_____

QA Management Approval: _____Date:_____

SOP Description

This Standard Operating Procedure (SOP) describes the process to be followed by TechLaw staff when acquiring analytical support. All requests for analytical services are to be arranged through the Laboratory Assistance Team (LAT) Coordinator, or alternatively through a LAT member (see Attachment A for a list of approved LAT members). This SOP is to be followed by the TechLaw project manager or designee when completing and submitting the Analytical Support Request Form (ASRF) (see Attachment B), and after the sampling event is completed by submitting copies of the chain-of-custody forms to the LAT and forwarding invoices to the project files. A LAT member checklist is provided in Attachment D and a TechLaw Project Manager checklist is provided in Attachment E.

This SOP is also to be followed by the LAT Coordinator and assigned LAT members when processing the ASRF, selecting a laboratory and reviewing data packages and invoices.

General Procedures

Related SOPs

This SOP is to be used in conjunction with other applicable SOPs found in the following SOP categories:

Category No.	Category Title
01	General Procedures
02	Field Procedures
03	Field Documentation Procedures
04	Packaging and Shipping Procedures
05	Field Equipment Operation and Maintenance Procedures
06	Groundwater Sampling/Monitoring and Analysis Procedures

07	Soil/Sediment Sampling and Analysis Procedures
08	Surface Water Sampling and Analysis Procedures
09	Health and Safety Procedures
10	Regulatory Compliance Procedures
11	Quality Assurance Procedures
12	Incineration/BIF Sampling and Analysis Procedures
13	Waste Sampling and Analysis Procedures
14	Asbestos Handling
15	Region 5 ESAT-Specific SOPs
16	Region 8 ESAT-Specific SOPs

Procedures for Submitting and Processing the Form

The ASRF (see Attachment B) must be submitted to the assigned LAT member at least five business days prior to the sampling event to avoid additional charges for rush shipping of any necessary supplies. If supplies are not required from the laboratory, the ASRF may be submitted at least three business days prior to the sampling event. If shorter turnaround time is required, every effort will be made by the LAT to process the request. Faxed, hand-written requests or electronic requests are acceptable as long as they are legible and complete.

The assigned LAT member will ensure that all necessary information is included in the ASRF, and make necessary arrangements with the laboratory (e.g., request delivery of glassware or other sample collection media and equipment). The assigned LAT member will procure sample containers on an as-needed basis, as indicated on the ASRF.

Upon completion of the ASRF by the assigned LAT member, a copy is uploaded to the project folder under LAT on SharePoint. All samples must be sent to a TechLaw-approved laboratory. The laboratory is selected on the ability to perform the requested analysis, availability of laboratory space, and analysis cost. A Work Authorization (Attachment C) is submitted to the selected laboratory by the assigned LAT member prior to sampling activities. The Work Authorization is also e-mailed to TechLaw contracting staff for generation of a purchase order (PO). A copy of the finalized Work Authorization is placed in the project folder under LAT on SharePoint.

Procedures for Completing the Form

The ASRF (see Attachment B) must contain complete sampling and analytical information. This information shall include the following:

- Project number (billing code) and project manager name;
- Site name and location;
- Date(s) of sampling event;
- Required turnaround time;
- Type of data package (i.e., Level II, III or IV, requirements for summary forms);
- Special considerations, if any; and
- For the table: sample matrix; number of field samples; parameter (i.e., the appropriate analytical method numbers); required detection limits; and the numbers of quality control (QC) samples (i.e., field duplicates, trip blanks, field blanks, and matrix spike/matrix spike duplicates [MS/MSD]).

Procedures for Changes

If changes occur in the number of samples or type of sampling methods during the field activities, the project manager will notify the LAT member by email.

Procedures After Sampling Event Completion

Chain-of-Custody (COC) Forms

Within one week of shipment of samples (within 24 hours ideally), the project manager must ensure that legible copies of all COC forms have been sent to the assigned LAT member. Faxes or scanned e-mail copies are acceptable.

Data Package Review and Delivery

The laboratory sends all data packages directly to the assigned LAT member, who performs a preliminary review to ensure that the laboratory has submitted the requested information.

This review shall be performed within approximately 1 day of receipt. Each data package is evaluated by the assigned LAT member for the following criteria ONLY:

- Laboratory reports address methods specified on COCs.
- Requested results present for all samples.
- Appropriate level data package provided.
- Data received within requested turnaround time.

Upon completion of this review, the original data package is forwarded to the project manager, unless the project manager specifically requests otherwise.

Invoices

The laboratory sends all invoices to the assigned LAT member. Upon receipt of the invoice, it is reviewed for agreement with the Work Authorization and project sampling documentation. Upon confirmation (within approximately 24 hours of receipt), the LAT member will forward the invoice to TechLaw Accounts Payable and send a copy to the TechLaw Project Manager. A copy of the approved invoice is uploaded by the LAT member to the company intranet site, under LAT > Project Files.

Communication with the Laboratories

A member of the LAT, preferably the assigned LAT member, is to participate in all communications with laboratories. This is to ensure that all procedures required under the Laboratory Agreements and the TechLaw Quality Assurance Program Plan are met. The LAT member will notify the laboratory of any unusual situations, including the expected presence of high concentrations of contaminants at the site.

Obtaining New Laboratories

All samples must be sent to a TechLaw-approved laboratory. Names of approved laboratories may be obtained from the LAT Coordinator. Only senior members of the LAT, with the concurrence of the TechLaw Quality Assurance Director (QAD), may approve a new laboratory; this approval is conditional upon examination of laboratory-specific documentation. Necessary

documentation consists of a laboratory quality assurance plan (must include a description and number of instruments, staff resumes and a summary of quality assurance/quality control procedures), SOPs, method detection limits (MDLs), quantitation limits, PE sample results and pricing.

A laboratory audit may also be performed by the TechLaw LAT. Audits will be performed for any laboratory that will be utilized on an ongoing basis. A laboratory audit may not be required if so directed by the client, or if only a specialty analysis is requested and the laboratory will not be used on a routine basis. The laboratory audit checklist is included as Attachment F to this SOP.

After a laboratory has been approved by the LAT, a Laboratory Agreement shall be arranged by the TechLaw Contracts Administrator, working in conjunction with the senior LAT member responsible for assessing the laboratory's qualifications. Only after these procedures have been completed may a new laboratory be used for analytical services.

The laboratory list is reviewed and updated annually, as necessary. LAT laboratories are contacted to obtain any updated laboratory documentation and pricing. Audits will be conducted at least every other year for laboratories performing routine analyses in the program.

Health and Safety

Not applicable

QA/QC

None at this time

Comments/Notes

Under no circumstances is it acceptable to provide the laboratory with the name, location or other identifying information for the site (this includes listing facility information on the chain-of-custody). Facility initials, TechLaw project number or other identifier should be used that will not reveal facility information to the laboratory, but will be evident to TechLaw employees involved with the project. If the laboratory becomes aware of the site name, the LAT member should inform the TechLaw COI Officer immediately. The COI Officer will ensure that the laboratory does not have a COI and will post documentation of this confirmation to the LAT project files on SharePoint.

The time required to arrange analytical services and process data packages and invoices will be charged to the appropriate project.

Attachments

Attachment A — Approved LAT Members

Attachment B — Analytical Support Request Form

Attachment C — Laboratory Work Authorization (generic)

Attachment D — LAT Member Checklist

Attachment E — TechLaw Project Manager Checklist

Attachment F — Laboratory Audit Checklist

References

TechLaw, Corporate Quality Management Plan, most current revision.

TechLaw, Health and Safety Program, most current version

TechLaw, Inc. Approved LAT Members

LAT Coordinator: Ms. Kim Whitlock
205 West Wacker Drive, Suite 1622
Chicago, IL 60606
(312) 345-8930
(217) 721-5483 (mobile)

LAT Members:

Ms. Rachel Ireland 7 Technology Drive, Unit 202 North Chelmsford, MA 01863 (978) 275-9749 (617) 283-1332 (mobile)	Ms. Zara Brown 14500 Avion Parkway, Suite 300 Chantilly, VA 20151 (703) 818-3213 (865) 898-3815 (mobile)
Mr. Gene Nance 5455 County Road 2 Chesapeake, OH 45619 (740) 867-0968 (304) 830-1442 (mobile)	Ms. Amy Dahl 1325 4th Ave, Suite 555 Seattle, WA 98101 (206) 826-5375 (206) 818-8891 (mobile)
Mr. Scott Walker 16194 W. 45th Dr. Golden, CO 80403 (303) 312-7726 (303) 453-9018 (mobile)	

If none of the above LAT Members are available, and an urgent laboratory need/data issue arises, please contact:

Ms. Jana Dawson
14500 Avion Parkway, Suite 300
Chantilly, VA 20151
(703) 818-3254
(703) 627-0821 (mobile)

Mr. Terry Zdon
1299 Washington Ave, Suite 270
Golden, CO 80401
(303) 552-5807
(773) 343-8974 (mobile)

TechLaw COI Officer: Ms. Judy Manley
14500 Avion Parkway, Suite 300
Chantilly, VA 20151
(703) 818-3233
(703) 209-5187 (mobile)

ANALYTICAL SUPPORT REQUEST FORM

Project Number (Bill Code): _____ **TechLaw Project Manager:** _____

Site Name and Location: _____

Site Code (identifier)*: _____

***Site Code should not reveal the site name or location and will be the only identifier used in communication with the laboratory (including COCs) to prevent COI issues**

Example: Site Name and Location: Buckeye Products in Adrian, Michigan
Site Code: BAM (used the first letter of facility name, city, and state)

Date(s) of Sampling Event: _____

Glassware: Date Needed: _____ **Location:** _____

Turnaround Time (circle one): Standard (21 days) / Rush-_____ days (extra charge)

Data Package: Level IV (full "CLP-like") / Other (Level II or III)_____

Electronic Date Deliverable? Yes / No Format (i.e., Excel): _____

Special Considerations:

- **Are any special certifications required?**
- **Are there minimum volume or filter requirements?**
- **Is there a specific QAPP requirement?**
- **Are high concentrations expected?**
- **Are verbal or preliminary results required?**

PLEASE ATTACH A TABLE OF THE APPLICABLE SCREENING LEVELS FOR ALL COMPOUNDS TO BE ANALYZED. The LAT staff will use this information to verify that the laboratory reporting limits will meet the specified screening levels.

Matrix [1]	Number of field samples	Parameter (method #'s)[2]	Required Reporting Limits	Number of field duplicates	Number of trip blanks	Number of field blanks	Number MS/MSD

1 Be specific (i.e., surface H2O, liquid fuel, slag; not just solid/water).
2 Be specific; if split sampling, attach applicable MDLs, action levels, methodologies, etc., from facility work plan. For metals, please specify which compound list should be utilized: RCRA 8, priority pollutants, Target Analyte List

Note: Target Analyte List compounds will be utilized, unless otherwise specified by the Project Manager.

TechLaw Project Manager Signature: _____ **Date**: _____
LAT Signature:_____ **Date:** _____

ATTACHMENT C [Revised 04/27/12]
SOP Number: 02-06-05

Date:

To: Contact
Laboratory
Address
Address

From: Kim Whitlock, LAT Coordinator
205 West Wacker Drive, Suite 1622
Chicago, IL 60606
(312) 345-8930
(312) 345-8979 (fax)

Re: EPA Prime Contract XXXXX
TechLaw Laboratory Agreement
Task Order Authorization Number: XXXX
Site Code (identifier): XXX
Project Code: [Insert billing code]

This document authorizes work on the subject Task Order as outlined in the attached Scope of Work and Pricing quotation (Attachment A). The expenditure limits on the Task Order are XXXXX. If it is anticipated that these funding limitations will be exceeded in performance of this work, you must notify us in a timely manner. Failure to notify and negotiate additional funding will result in forfeiture of costs incurred in excess of the funding limitations. Invoices should be sent to the Laboratory Assistance Team (LAT) representative noted above.

Please acknowledge your acceptance of work by signing in the space provided on the form, faxing a signed copy of this Task Order Authorization Form to Brenda Smith at (703) 818-8813, and returning the original within 5 days of your receipt to [LAT member name]. By acceptance of this Work Authorization, the Laboratory confirms that: no known personal or organizational conflict of interest exists; best efforts will be employed to conduct the work specified to the satisfaction of TechLaw, Inc. representatives; all terms and conditions of the Agreement identified and the Scope of Work and Pricing document will be met in performance of the work specified herein.

Authorized Signatures:

TechLaw, Inc.	**Laboratory Name**
Name:	Name:
Title:	Title:
Date:	Date:

ATTACHMENT D [Revised 04/27/121]
SOP Number: 02-06-05

LAT Member Checklist

1. If a TechLaw project manager contacts you regarding laboratory procurement, please ask them to fill out the Analytical Support Request Form (ASRF) in Attachment B of SOP 02-06-05, if they have not already done so. Upload completed form to the project file on SharePoint.
    a. Be sure to ask if there are any reporting limit requirements, and verify with the laboratory that they can achieve such requirements
2. Contact three TechLaw Approved Laboratories to obtain price quotes and to ensure they have capacity to analyze the samples within the requested turnaround time.
    a. If a specialized analysis is required and is not performed by a TechLaw Approved Laboratory, another laboratory may be used upon consultation with the LAT Coordinator.
3. Select the laboratory based on lowest pricing and ability to perform the requested analyses.
4. Fill out the Work Authorization Form in Appendix C of SOP 02-06-05, PDF file, and attach the analytical quote to end of the PDF. Submit the form to the selected laboratory via e-mail (copy Ms. Brenda Smith and Ms. Judy Manley). Ms. Smith will email the LAT member the PO associated with the analytical request.
5. Check the appropriate laboratory folder under LAT on SharePoint to ensure we have the SOP for the methods requested for the project. If these are not available already on SharePoint, request a copy of the SOP from the lab and upload to the laboratory folder on SharePoint.
6. Create a folder for your project under LAT > Project Files.
7. Update the Project Tracking spreadsheet under LAT > Project Tracking and ensure justification for laboratory selection is included in the Comments column.
8. Upon submission to the laboratory, upload a copy of the Work Authorization Form (unsigned by lab) to LAT > Project Files > Project Name.
9. Once a signed copy of the Work Authorization Form is received from laboratory, upload to LAT > Project Files > Project Name.
10. Confirm with laboratory when/where to send bottleware.
11. Inform the laboratory when to expect samples (it is also a good idea to remind them one day before they will receive samples).
12. Ensure the TechLaw field team submits a copy of the COC to you for verification of invoicing/sample data.
13. Upon receipt of the analytical data, review for the following:
    a. Laboratory reports address methods specified on COCs
    b. Requested results present for all samples submitted to the lab
    c. Appropriate level data package provided
    d. Data received within the requested turnaround time
    e. Scan through the laboratory case narrative for any major issues that would result in rejection of data.
14. If the items above are acceptable, approve invoice by signing, dating, and adding the proper PO number (including line item number) and forward to Patti Pinkard.
15. Upload a copy of the approved invoice to the project folder under LAT > Project Files.
16. Email a copy of the approved invoice to the TechLaw project manager.
17. Ask the TechLaw Project Manager where the data should be sent, and send out ASAP. If electronic data is available, upload a copy to the project folder under LAT > Project Files

ATTACHMENT E [Revised 04/27/12]
SOP Number: 02-06-05

TechLaw Project Manager Checklist for LAT

1. Fill out the Analytical Support Request Form in Attachment B of SOP 02-06-05.
2. Notify LAT member of any changes in schedule/requirements as soon as possible.
3. Ensure Field Team provides a copy of completed COCs to LAT member.
4. Inform LAT member where to send data upon receipt.

LAT members will:

- Procure laboratories
- Order bottleware, preservatives, and laboratory-grade water for blanks (if requested to do so)
- Handle all communication with the laboratory
- Add another laboratory to the TechLaw Approved Lab List only if required by the project (i.e., 24-hour turnaround for an emergency response) and current approved laboratories cannot fulfill the project requirements. Note: Additional time will be required to obtain the necessary information and add the laboratory to the approved list.

LAT members do NOT:

- Verify reporting limits if no requirements have been provided by the TechLaw Project Manager
- Perform data validation (unless qualified and asked to do so)
- Coordinate a data validator (unless asked to do so and authorized hours are provided)

LABORATORY AUDIT CHECKLIST

Laboratory On-Site Visits

There are several purposes for making on-site visits to analytical laboratories. The most common purposes are:

1. Prior to award of a contract or delivery of samples, a client visits the laboratory to verify that the laboratory has the capability to perform the needed work. The areas for which capability must be judged are:

    - Physical facility – adequate work space, adequate and appropriate air handling, adequate storage space.
    - Equipment – all equipment (instrumentation, reagents, glassware, etc.) needed to do the job at the needed frequency.
    - Personnel – trained, experienced personnel who meet the clients' requirements.
    - Standard Operating Procedures (SOPs) – written procedures must be in place (and updated when changes to "modus operandi" are made) for all operations of the laboratory so that consistency and continuity are maintained where appropriate.
    - Quality Assurance Program – includes all aspects of EPA's "Good Automated Laboratory Practices" (GALP) Guidance.
    - Appropriate evidentiary procedures, Chain-of-Custody documentation, and security systems must be in place.

2. Post contract award or after sample delivery by a client (at intervals determined appropriate by the client), the laboratory can be visited to verify that the capabilities evaluated in Number one (1) still exists, or improvements cited as needed or deficiencies cited as requiring correction in Number one (1) have occurred.

3. Problem resolution visits – When problems are noted by the client (e.g., performance evaluation samples not analyzed acceptably, lateness, non-compliance with contract requirements, etc.) laboratories can be visited to isolate problem areas and identify where corrective action must be taken by laboratory management.

4. Unannounced visits – to verify that the laboratory follows procedures and maintains systems per client's requirements, even when the client's visit is not expected.

5. Unannounced visits – when there is reason to believe a laboratory may be involved in improper practices (e.g., data falsification/alteration), a client may want a "surprise" visit. This visit should focus an audit on the area perceived to be vulnerable.

6. Routine – even when a laboratory is performing well, a client presence to show interest (and maybe a "pat on the back") is important every other year at a minimum.

As we look at the cited purposes, we should take the opportunity to identify what we really need to do during an on-site visit to meet our needs. We should minimize the universe of possible targets for evaluation and focus on what is important.

We should recognize that the capability to meet our needs (produce our required product) may come in many forms, and a stereotyped approach on our part is unreasonable and unnecessary. If we accept this premise, we go a long way towards minimizing our efforts in auditing the laboratory and opening the door to innovation and creativity on the part of our laboratories that may save time and money and may produce a better product.

Since the first two types (and sometimes Number 6) of on site visits are the most common and involve looking at the same things (which are primarily amenable to a checklist approach), the first effort at a new design of an on-site visit will consist of an appropriate checklist.

It is important to note that a checklist only meets part of our needs: one-on-one conversations with laboratory personnel who will perform our work should occur to make sure they understand our requirements, follow their SOPs, are properly trained, etc.

Proposed checklists for Technical and Evidentiary on site visits (audits) are attached. They are designed to be used together so one auditor can perform the full gamut of evaluation in one swing through a laboratory. The auditor should become familiar with the checklists prior to an audit so that all necessary checks can be made in one location in a laboratory at one time.

If items (e.g., SOPs) are addressed in the evidentiary section, then they are not addressed in the technical section. Since there is so much in common for organics and inorganics, the checklist is combined, with items unique to one or the other clearly identified.

These checklists are not contract specific. They can be used for non-CLP and CLP because they do not demand conformance, only observance of what is in place. The auditor is responsible for determining if the laboratory appears to meet the requirements of the client (which in the case of CLP labs, is the SOW).

The auditor should only evaluate the laboratory according to the items that the client considers relevant to meet needs (for CLP contracts, this is all items). Judgment should be used when determining what items are relevant to meet needs.

Laboratory On-site Visit Evaluation

Laboratory name:

Address:

Telephone number:

Date of in-site visit:

Type of evaluation 1) organics 2) inorganics 3) evidentiary

Evaluation Team Members

Name	Title	Evaluated 1, 2 or 3 above?

Personnel interviewed/contacted

Name	Title	Interviewed for 1, 2 or 3 above?

Evidentiary Procedures Evaluation Checklist

I. Sample Receipt

	Check one Yes	No
1. Is there a designated sample custodian and alternate for each shift?	_____	_____

Names:

Sample custodians	Alternates
_____	_____
_____	_____

2. Are standard operating procedures (SOPs) for sample receipt in place and readily available?	_____	_____
3. Are SOPs for sample receipt followed by laboratory personnel?	_____	_____
4. Is the sample receipt area secured against non-authorized personnel?	_____	_____
5. Does sample custodian verify the following:		
a. Condition of shipping cooler	_____	_____
b. Presence or absence of custody seals	_____	_____
c. Condition of custody seals, when present	_____	_____
d. Custody seal numbers, when present	_____	_____
e. Presence or absence of chain-of-custody record(s)	_____	_____
f. Presence or absence of airbill stickers	_____	_____
g. Airbill or airbill sticker number	_____	_____
h. Presence or absence of sample tags	_____	_____
i. Sample tag numbers (if applicable)	_____	_____
j. Condition of sample containers	_____	_____
k. Discrepancies in any information recorded on chain-of-custody records, client requests, airbills, sample containers, etc.	_____	_____

	Yes	No
l. Documentation of hand deliveries	_____	_____
m. Problems encountered	_____	_____
6. Obtain examples of all forms used during sample receipt	_____	_____
II. Sample Identification		
1. Does the laboratory have a unique sample identification system (i.e., vs. using client sample identification numbers)?	_____	_____
a. Is the number assigned upon receipt? If no when?_____	_____	_____
b. If "yes", are numbers cross referenced to client numbers in a log?	_____	_____
2. Does the system clearly apply to samples, extracts, digestates, etc.?	_____	_____
3. Are SOPs readily available for sample identification?	_____	_____
4. Are SOPs for sample identification followed by laboratory personnel?	_____	_____
5. Obtain example of laboratory's sample identification system (e.g., example sample number with cross reference)	_____	_____
III. Sample Storage and Tracking		
1. Are sample (and extracts, etc.) storage areas secured and access to samples (extracts, etc.) available only to authorized personnel?	_____	_____
2. Are samples (extracts, etc.) logged in/out of storage area(s) when accessed?	_____	_____
3. Are samples (extracts, etc.) tracked throughout analytical process (e.g., a traveler sheet)?	_____	_____
4. Are SOPs for storage and tracking of samples (extracts, etc.) readily available?	_____	_____

	Yes	No
5. Are SOPs for storage and tracking followed by laboratory personnel?	_____	_____
6. Obtain examples of all forms/documents used for storage and tracking records	_____	_____
IV. Document Review		
1. Evaluate documents with the following questions in mind.	_____	_____
a. Are activities (e.g., GC/MS-VOA, ICP-metals) identified on all analysis documents?	_____	_____
b. Are titles on all documents?	_____	_____
c. Are columns labeled with headers?	_____	_____
d. Are reviewers' signatures identified when applicable?	_____	_____
e. Is the laboratory's name on all documents?	_____	_____
f. Are all entries fully dated (day, month, year)?	_____	_____
g. Are entries signed by the responsible person for performing and recording activities?	_____	_____
h. Are all logbook and other document entries in ink?	_____	_____
i. Is error correction protocol followed? (single line through area to be corrected and corrector's initials – no "white out")	_____	_____
j. Are pages in bound and unbound logbooks sequentially numbered?	_____	_____
k. Are log-book entries in chronological order?	_____	_____
l. Is inserted information taped into logbooks signed and dated when activity is performed?	_____	_____
m. Are unused portions of documents lined out?	_____	_____

	Yes	No
2. Do documents provide a complete record of activities observed by the evaluator?	_____	_____
3. Are instrument run logs maintained to enable a reconstruction of the run sequence on an instrument?	_____	_____
4. Are records of failed runs maintained?	_____	_____
5. Is disposal/depletion of samples (extracts, etc.) documented?	_____	_____
6. Is data transferred electronically within the laboratory? If "yes", is a hard copy printed and retained in a client/case file?	_____	_____
a. If data is transferred electronically, is the following information recorded?	_____	_____
1) Person responsible for electronic data transfer?	_____	_____
2) Date of electronic transfer?	_____	_____
3) Person to whom data was electronically transferred?	_____	_____
4) Status of electronically transferred data (e.g., draft final, etc.)?	_____	_____
5) Numerical identifier assigned to electronic data transfer?	_____	_____
b. Are SOPs readily available for electronic data transfer?	_____	_____
c. Are SOPs followed by laboratory personnel for electronic data transfer?	_____	_____
V. Confidential Information		
1. If laboratory receives confidential information/documents, is a system set up to maintain that confidentiality, including for data generated on associated samples?	_____	_____

VI. Case (Client's Designated Group of Samples) File Organization and Assembly

Name:_____ _____
Document Control Officer Alternate

		Yes	No
1.	Are case documents maintained in a secure area?	_____	_____
2.	Is shipment of deliverables to clients documented?	_____	_____
	a. Is recipient identified?	_____	_____
3.	Are deliverables sealed with custody seals?	_____	_____
	a. Are custody seals signed?	_____	_____
	b. Are custody seals dated?	_____	_____
4.	Does document control officer assemble and cross check information to assure that data on each case file is consistent and complete?	_____	_____

VII. Security of the Facility

1.	Are visitors required to sign in?	_____	_____
2.	Are visitors required to display distinct badges/ID?	_____	_____
3.	Are all doors to outside locked except to reception area?	_____	_____
4.	Is access to laboratory and data reduction/report preparation areas limited to authorized personnel?	_____	_____

Observations/Comments by Evaluators

I Sample receipt –

II Sample identification –

III Sample storage/tracking –

IV Document review –

V Confidential information –

VI Case file organization and assembly –

VII Security of facility –

Technical Procedures Evaluation Checklist
(Organics and Inorganics)

		Yes	No
I.	Sample Receipt and Storage Area (Evaluate with I-III of Evidentiary Audit)		
1.	Are sample shipping coolers opened in a contamination-free area (e.g., fume hood or vented area)?	_____	_____
2.	Are adequate facilities provided for the cold storage of samples and unused samples for 60 days after data submission?	_____	_____
a.	Is the temperature of the cold storage recorded daily in a logbook?	_____	_____
b.	Are temperature excursions noted and appropriate actions taken when required? (Check SOP)	_____	_____
3.	For inorganic only: Is the pH of the samples recorded and available for the data review?	_____	_____
4.	For organic only: Are volatile samples stored separately from semi-volatile samples and extracts?	_____	_____
5.	For organic only: Are VOA holding blanks present in the volatile sample storage facility? (One per case)	_____	_____
6.	For organic only: Are sample extracts properly stored (2-6oC, separate) and easy to locate by reference to a logbook?	_____	_____
II.	Sample Preparation Area		
1.	Is the laboratory maintained in a clean and organized manner appropriate for trace level analyses (contamination free)?	_____	_____
2.	Does the laboratory appear to have adequate work space (6 linear feet or unencumbered bench top/analyst)?	_____	_____

		Yes	No
3.	Are laboratory benches made of suitable chemically resistant materials?	_____	_____
4.	Are sufficient functional hoods available?	_____	_____
5.	Is documented organic free water (for organics standards, blanks, dilutions) or distilled/ demineralized water (for inorganics) available?	_____	_____
6.	Are analytical balances located away from drafts and areas subject to rapid temperature changes?	_____	_____
	a. Are the balances checked routinely (e.g., before each weighing session) with the appropriate range of weights and results recorded in a permanent notebook?	_____	_____
	b. Are routine weights checked against class S weights at least once a month and Results recorded in a permanent notebook?	_____	_____
	c. Have the balances been calibrated within one year by a certified technician?	_____	_____
	d. Are the data generated from balances electronically transferred or manually entered into LIMS?	_____	_____
7.	Are sample preparation SOPs readily available?	_____	_____
	- Are sample preparation SOPs followed by laboratory personnel?	_____	_____
8.	Are glassware preparation/cleaning SOPs readily available?	_____	_____
	- Are they followed by laboratory personnel?	_____	_____
9.	Is all required sample preparation equipment available?	_____	_____
	For organics only:		
	a. Sonicator	_____	_____

Make_____Model_____Backup_____(Y/N)

	Yes	No
b. GPC	_____	_____
Make_____Model_____Backup_____ (Y/N)		
c. GPC UV Detector	_____	_____
Make_____Model_____		
d. Do GPC logs indicate corrective actions are taken when there is a problem with calibration?	_____	_____
e. Continuous liquid/liquid extractors? Number_____	_____	_____
For inorganics only:		
f. If microwave digestion is to be used, is an adequate microwave ovens (programmable power setting up to 600 watts) available?	_____	_____
10. Do analysts record bench data in a neat and accurate manner?	_____	_____
11. Do analysts record lot number of solvents, spiking solutions, etc., on bench sheets?	_____	_____
12. Is there evidence of a secondary review of all documents and logbooks by someone other than the person generating the documents?	_____	_____
13. Review the following procedures for oven drying:		
a. Are the temperatures in the drying ovens verified against NIST traceable thermometers?	_____	_____
b. Do ovens have temperature logbooks?	_____	_____
c. Are “in” & “out” drying times recorded?	_____	_____
III. Standards Preparation and Storage		
1. Are SOPs for standards preparation readily available?	_____	_____

		Yes	No
2.	Are SOPs followed by laboratory personnel?	_____	_____
3.	Are reagent grade or higher purity chemicals used to prepare standards?	_____	_____
4.	Are standards properly labeled with concentrations, date of preparation, expiration date, and/or a traceable reference code number?	_____	_____
5.	Are spiking/calibration standards preparation and tracking logbooks maintained for:	_____	_____
	Inorganics?	_____	_____
	Semi-volatiles?	_____	_____
	Pesticides?	_____	_____
	Volatiles?	_____	_____
	- Are logbook numbers and series of stock Solutions and reagents recorded?	_____	_____
6.	If the laboratory purchases commercially prepared standard mixes, is appropriate documentation available? (Manufacturer's Certificate of Analysis)	_____	_____
7.	Inorganic only: If the laboratory uses automatic pipets for preparing standards, are they routinely calibrated?	_____	_____

IV. Analytical Instrumentation and Analyses-Specific Items

ORGANICS

A.	GC/M's (for up 200 samples/month)	_____	_____
1.	Instrument needs	_____	_____
	a. 1 VOA GC/MS/DS with purge and trap device	_____	_____
	b. 2 semi-VOA GC/MS	_____	_____
	c. 1 Backup GC/MS/DS and purge and trap device	_____	_____

GC/MS/DS Instr ID	Manuf./model	Software rev.	Purge and Trap InstrID	Manuf/model

		Yes	No
2.	Are manufacturers' operating manuals readily available?	_____	_____
3.	Does laboratory have service contracts?	_____	_____
	a. Does lab have extensive replacement parts available?	_____	_____
4.	Is a permanent service record maintained for each instrument?	_____	_____
5.	Does the laboratory use a recent mass spectral library?	_____	_____
6.	Magnetic tape storage of GC/MS electronic data:	_____	_____
	a. Are raw data, including quantitative output files and libraries, archived on magnetic tape?	_____	_____
	b. Is a log of raw data contents of tapes maintained?	_____	_____
7.	VOA analyses:	_____	_____
	a. Is equipment available for heated purge and trap for low level soil analysis?	_____	_____
	b. Are VOA holding blanks results available?	_____	_____
8.	Can instrument operator show from the run log that corrective actions have been taken for:	_____	_____

		Yes	No
	a. Re-analyses when internal standard areas are out of control?	_____	_____
	b. Dilutions when calibration range is exceeded?	_____	_____
	c. Blanks when previous sample showed saturation?	_____	_____
9.	Are SOPs readily available for GC/MS analyses and logbook completion?	_____	_____
	Are they followed by the laboratory?	_____	_____
10.	Is there evidence of a secondary review of all documents and logbooks by someone other than the person generating the documents?	_____	_____
B.	GC/EC's (for up to 200 samples/month)	_____	_____
1.	Instrument needs	_____	_____
	a. 2 GC/EC/DS with dual columns	_____	_____
	b. 1 backup GC/EC	_____	_____

GC/EC			Data System	
Instr ID	Manuf./Model	Detector	Manuf./Model	Revision #

2.	Are manufacturers' operating manuals readily available?	_____	_____
3.	Does the laboratory have service contracts?	_____	_____
	a. Does the lab have extensive replacement parts available?	_____	_____
4.	Is a permanent service record maintained for each instrument?	_____	_____

		Yes	No
5.	Are SOPs readily available for GC/EC analyses and logbook completion?	_____	_____
	a. Are they followed by laboratory personnel?	_____	_____
6.	Is there any evidence of a secondary review of all documents by someone other than the person generating the document?	_____	_____

INORGANICS

A. ICPs (for up to 300 samples/month)

1.	Instrument needs	_____	_____
	a. 1 ICP	_____	_____

Instrument ID Manuf./Model Seq./Sim

_____
_____
_____
_____

		Yes	No
2.	Are manufacturers' operating manuals readily available?	_____	_____
3.	Does laboratory have service contracts?	_____	_____
4.	Are SOPs readily available for ICP analyses and logbook completion?	_____	_____
	Are they followed?	_____	_____
5.	Are stock standards current?	_____	_____
6.	Are calibration standards made from a ready made stock standard? Manufacturer?_____	_____	_____
	Are they prepared at least monthly?	_____	_____

		Yes	No
7.	Have any of the instruments been modified?	_____	_____
	If "yes", which one and how?_____		
8.	How will calibration intensity and gains be kept?_____		
9.	Is a mass flow controller used?	_____	_____
10.	Is interference correction done automatically and are interelement correction factors determined on at least an annual basis?	_____	_____
11.	Is a permanent service record maintained for each instrument?	_____	_____

B. ICP-MS (for up to 300 samples/month)

1.	Instrument needs	_____	_____
	a. 1 ICP-MS	_____	_____

Instrument ID Manuf./Model Seq./Sim

_____

_____

_____

_____

2.	Are manufacturers' operating manuals readily available?	_____	_____
3.	Does laboratory have service contracts?	_____	_____
4.	Are SOPs readily available for ICP-MS analyses and logbook completion?	_____	_____
	Are they followed?	_____	_____
5.	Are stock standards current?	_____	_____
6.	Are calibration standards made from a ready made stock standard? Manufacturer?_____	_____	_____
	Are they prepared at least monthly?	_____	_____

		Yes	No
7.	Have any of the instruments been modified?	_____	_____
	If "yes", which one and how?_____		
8.	How will calibration intensity and gains be kept?_____		
9.	Are reanalysis performed when internal standards are out of control?	_____	_____
10.	Is a mass flow controller used?	_____	_____
11.	Is interference correction done automatically and are interelement correction factors determined on at least an annual basis?	_____	_____
12.	Is a permanent service record maintained for each instrument?	_____	_____

C. Atomic Absorption (AA) Spectrometer

1.	Instrument needs		
	a. 2 GFAA for up to 200 samples/month	_____	_____
	b. 4 GFAA for up to 400 samples/month	_____	_____

GFAAs

Instrument ID	Manuf./Model

2.	Are element specific SOPs which list instrument conditions, background corrections, and required instrument sensitivity readily available?	_____	_____
	Are they followed?	_____	_____
3.	Are calibration results (i.e., sensitivity) kept in a permanent record to track instrument performance?	_____	_____

		Yes	No
4.	Are calibration standards prepared daily?	_____	_____
5.	Have any of the instruments been modified?	_____	_____
	If "yes", which ones and how?_____ _____		
6.	Is an autosampler used?	_____	_____
7.	Are EPA or instrument manufacturers matrix modifiers used?	_____	_____
	As: _____ Pb: _____ Se: _____ Tl: _____		
8.	Is a permanent service record maintained for each instrument?	_____	_____

D. Mercury analyzer – Cold Vapor AAs

1.	Instrument needs.	_____	_____
	a. 2 mercury cold vapor AAs for up 300 samples/month.	_____	_____
	Cold Vapor AA Instrument ID's Manuf./Model _____ _____ _____		
2.	Are SOPs readily available for analyses and logbook completion?	_____	_____
	Are they followed?	_____	_____
3.	Are calibration standards prepared daily?	_____	_____
	With the samples?	_____	_____
4.	Is an absorbance record kept to monitor sensitivity?	_____	_____
5.	Is a permanent service record maintained for each instrument?	_____	_____

E. Cyanide Distillation Apparatus _____ _____

1. Needs – 12 distillation apparatuses plus one photometer for up to 300 samples/month _____ _____

Photometer

Instrument ID's Manuf./Model

_____

_____

_____

	Yes	No
2. Are SOPs readily available for cyanide analysis?	_____	_____
Are they followed?	_____	_____
3. Is there a stock cyanide standard from a commercial source?	_____	_____
If "no", made from KCN salt?	_____	_____
Is standard titrated?	_____	_____
4. Is the titrimetric manual or semi-automated colorimetric method used? Method:_____	_____	_____
5. Is the pH of the samples recorded and available for review?	_____	_____
6. Are samples checked for the presence of sulfide and chlorine?	_____	_____
7. Is a service log maintained for the photometer?	_____	_____
V. Data Handling and Review (GALP)	_____	_____
1. Are data calculations spot checked by a second person?	_____	_____
2. Do records indicate appropriate corrective action when QC criteria are not met?	_____	_____
3. Do supervisory personnel review the data and QC results prior to submission?	_____	_____
4. Are SOPs for data handling/review readily available?	_____	_____
Are they followed?	_____	_____

		Yes	No
5.	Are data and file access user ID of file password protected?	_____	_____
6.	Are deliverables checked for completeness and accuracy? (Hard copy and electronic)	_____	_____
Resubmittals?		_____	_____
7.	Is the monthly data entry error rate determined and recorded?	_____	_____
8.	When changes to deliverables are required, are the changes properly documented? (Rationale, review, initials.)	_____	_____
9.	Are user manuals and operations/systems manuals available?	_____	_____
10.	Is a written software test and acceptance plan available for installation of system changes?	_____	_____
VI.	Quality Assurance Internal Inspections		
1.	Is there an internal QA inspection procedure?	_____	_____
2.	Does the QA officer report to senior management?	_____	_____
3.	Are corrective actions documented?	_____	_____
4.	What kinds of internal audits are performed?	_____	_____
	a. Blind PE sample?	_____	_____
	b. Other:_____ _____	_____	_____
5.	What kinds of QA records are kept?		
	a. PE sample results?	_____	_____
	b. Records of recoveries (extractions, etc.)	_____	_____
	c. Training/experience records of personnel?	_____	_____
	d. Method sensitivities?	_____	_____

		Yes	No
	e. Control charts for QC purposes?	_____	_____
	f. Other_____		
VII.	Quality Assurance Plan (QAP)		
	1. Is a QAP available?	_____	_____
	2. Does it address the following?	_____	_____
	a. Organization and philosophy	_____	_____
	b. Facilities and equipment	_____	_____
	c. Document control	_____	_____
	d. Analytical methodology	_____	_____
	e. Data generation	_____	_____
	f. QA	_____	_____
	g. QC	_____	_____
	h. Corporate ethics policy	_____	_____
VIII.	Standard Operating Procedures		
	1. Are SOPs available for the following (many already addressed earlier during the audit)?	_____	_____
	a. Evidentiary	_____	_____
	b. Sample Receipt and storage	_____	_____
	c. Sample preparation	_____	_____
	d. Glassware cleaning	_____	_____
	e. Calibration (balance)	_____	_____
	f. Calibration (instruments)	_____	_____
	g. Analytical procedures (for each system)	_____	_____
	h. Maintenance activities (for each system)	_____	_____
	i. Analytical standards	_____	_____
	j. Data reduction procedures	_____	_____
	k. Documentation policy/procedures	_____	_____
	l. Data validation/self inspection procedures	_____	_____
	m. Data management and handling	_____	_____

		Yes	No
IX.	Organization and Personnel Summary (See "Key Personnel" list – attached)	_____	_____
1.	Do personnel assigned to this project have the appropriate educational background to accomplish the objectives of the program?	_____	_____
2.	Is the organization adequately staffed to meet project commitments in a timely manner?	_____	_____
3.	Were all key personnel available? List those not present.	_____	_____

Name	Position

X. Laboratory Capacity

1.	Does the laboratory have sufficient analytical instrumentation to analyze the needed number of samples?	_____	_____
2.	Does the laboratory have sufficient technical administrative personnel to deliver the number of needed analyses?	_____	_____
3.	Does the laboratory have an adequate sample and data tracking system to handle the needed number of analyses?	_____	_____

XI. Summary

Provide an overall evaluation of the laboratory's apparent technical capability to perform the needed work.

KEY PERSONNEL LIST

List the personnel assigned to the following functional positions, and put a check next to any unqualified personnel.

Laboratory Operations Personnel

Project Manager

Name:_____
Responsible for overall aspects of contract/project: primary contract

Quality Assurance Officer

Name:_____
Generally requires a Bachelor's degree in chemistry/science/engineering + 3 years' laboratory experience, including 1 year applied experience with QA principles and practices in an analytical laboratory.

Laboratory Manager

Name:_____
Responsible for overall management of the laboratory.

Sample Custodian

Name:_____

Laboratory Personnel – Organics

Organics Supervisor

Name:_____
Generally requires Bachelor's degree in chemistry/science/engineering + 3 years' organics experience, including 1 year supervisory experience.

Sample Preparation Laboratory Supervisor

Name:_____
Generally requires Bachelor's degree in chemistry/science/engineering + 3 years' laboratory experience including 1 year supervisory experience. Three additional years experience may substitute for education requirement.

GC/MS Operator
Name:_____
Name:_____
Name:_____
Generally requires Bachelor's degree in chemistry/science/engineering + 1 year GC/MS/DS experience or 3 years GC/MS/DS experience and GC/MS interpretation. Three additional years experience may substitute for education requirement.

GC/EC Operator

Name:_____
Name:_____
Name:_____
Generally requires Bachelor's degree in chemistry/science/engineering + 1 year GC/EC experience or 3 years GC/EC experience and GC/EC interpretation. Three additional years experience may substitute for education requirement.

Extraction Concentration Expert

Name:_____
Name:_____
Generally requires High School diploma and college level course in general chemistry + 1 year experience in extraction/concentration.

Backup Chemists (Technical Staff Redundancy)

Name:_____
Generally requires Bachelor's degree in chemistry/science/engineering + 1 year lab experience in GC/MS operation, MS interpretation, extraction, and pesticide analysis.

Comments:

Laboratory Personnel – Inorganics

Inorganic Laboratory Supervisor

Name:_____
Generally requires a BS or BA in science. 1 year related experience, including 1 year as a supervisor.

ICP/ICP-MS Operator

Name:_____
Name:_____
Generally requires a BS or BA in science, 1 year ICP experience, 3 years additional experience in lieu of the education requirement.

Lachat Operator

Name:_____
Name:_____
Generally requires a BS or BA in science, 1 year Lachat experience, 3 years additional experience in lieu of the education requirement.

AA/Mercury Operator

Name:_____
Name:_____
Generally requires a BS or BA in science, 1 year experience for each of the following AA techniques: flame, graphite furnace, and cold vapor.
3 years additional experience in lieu of the education requirement.

Inorganic Sample Preparation Specialist

Name:_____
Name:_____
High school diploma and college level course in chemistry and 1 year related experience: 6 months experience plus 6 months experience with microwave digestion, if microwave technique is used.

Wet Chemistry Analyst

Name:_____
Name:_____
Generally requires a BS or BA in science and 1 year experience; 3 additional years experience may substitute for education requirement.

Attachment A
Black Chain of Custody Form

TechLaw
ESAT Region 8 Laboratory
16194 W 45th Drive
Golden, CO 80403
303.312.7047

US EPA CLP
Chain-of-Custody

EVENT: eCOC Template

Sample #	Tag	Location	Sub Location	Sample Type	Collection	Matrix	Analyses	Preservation	Sample Date	Sample Time	Remarks	Sampler

Relinquished By (DATE/TIME):

Received By (DATE/TIME):

Sampling/Analysis Notes:

Cooler Temp:_____
ICE: Y N
pH: Y N
Cust. Seals: Y N
COC/Labels Agree: Y N
Containers Intact: Y N

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Sampling/Analysis Notes:

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